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REVISION 0

REMOVAL ACTION SAMPLING AND ANALYSIS PLAN

FOR

CONFIRMATION SAMPLING OF SOIL
AND
PERIMETER AND PERSONAL SAMPLING OF AIR
FOR ASBESTOS

OPERABLE UNIT 02
FORMER SCREENING PLANT AREA
NEAR LIBBY, MONTANA

July 2000

DRAFT

Contract No. DTRS57-99-D-00017
Task Order No. C0006

Prepared for:

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LIST OF ACRONYMS AND ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
AHERA	Asbestos Hazard Emergency Response Act
AIHA	American Industrial Hygiene Association
ASTM	American Society for Testing and Materials
CDM	Camp Dresser & McKee Inc.
CDM Federal	CDM Federal Programs Corporation
CFR	Code of Federal Regulations
COC	Chain-of-Custody
DI	deionized
DQO	data quality objective
EMSL	EMSL Analytical Inc.
EPA	U.S. Environmental Protection Agency
° F	degrees Fahrenheit
f/cc	fibers per cubic centimeter
f/ml	fibers per milliliter
GPS	Global Positioning System
HEPA	Highly Efficient Particulate Air Filter
ISSI	ISSI Consulting Group, Inc.
ISO	International Standards
IRIS	Integrated Risk Information System
L/min	liters per minute
MARCOR	MARCOR Remediation, Inc. (subcontractor to CDM Federal)
μm	micron
MFL	million fibers per liter
mm	millimeter
NESHAP	National Emission Standards for Hazardous Air Pollutants
NIOSH	National Institute of Occupational Safety and Health
OSHA	Occupational Safety and Health Administration
OW	Office of Water, U.S. EPA
PCM	Phase Contrast Microscopy
PEL	permissible exposure limit
PES	Pacific Environmental Services, Inc. (subcontractor to CDM Federal)
PLM	Polarized Light Microscopy
QC	Quality Control
s/mm^2	structures per square millimeter
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
STEL	short term exposure limit
TEM	Transmission Electron Microscopy
TLV	Threshold Limit Value
TWA	time-weighted average

LIST OF ACRONYMS AND ABBREVIATIONS (CONTINUED)

USACOE	U.S. Army Corps of Engineers
VOC	volatile organic compound
Volpe Center	John A. Volpe National Transportation Systems Center

1.0 INTRODUCTION

The Environmental Engineering Division (DTS-33) of the John A. Volpe National Transportation Systems Center (Volpe Center) is providing environmental engineering and contaminant removal support for the Libby Asbestos Project to Region 8 of the U.S. Environmental Protection Agency (EPA). The Volpe Center and their contractor, CDM Federal Programs Corporation (CDM Federal) and its subcontractor, Pacific Environmental Services, Inc. (PES), along with the Volpe Center's removal/demolition contractor, MARCOR Remediation, Inc. (MARCOR), have been requested by EPA to provide the necessary technical support, equipment, and other services to implement and complete a time-critical removal action at Operable Unit 02, the Screening Plant Area formerly operated by W.R. Grace Company near Libby, Montana. The Volpe Center is functioning as coordinator of this effort, consolidating information provided by CDM Federal, PES, and MARCOR.

This document serves as the Sampling and Analysis Plan (SAP) for confirmation sampling of soil, and perimeter and personal air for asbestos for the Operable Unit 02 removal action. This SAP defines sampling and analytical procedures to be used for conducting the soil and air sampling at this site.

1.1 SITE BACKGROUND

The town of Libby is located in the extreme northwest corner of Montana. According to historical mining records, up to 80 percent of the world's vermiculite has come from the W.R. Grace Vermiculite Mine located on Zonolite Mountain located approximately seven miles northeast of Libby. Vermiculite is a mineral that is used in various building materials and textiles. Disseminated within the enormous deposit of vermiculite on Zonolite Mountain is the mineral tremolite, a rare and exceedingly toxic form of asbestos. Over the roughly 60-year life of the mine, tremolite asbestos was released into the environment and potentially in the town of Libby as a by-product of the mining and ore-processing activities. The Zonolite Mine began operation in 1924 by owner Edward Alley. In 1925, Great Northern Railroad shipped the first boxcar of "Zonolite" from Libby to an Ohio company that used it to insulate bank vaults, office safes, and filing cabinets. Other firms used the material to make building boards and roofing materials.

Processing the material was straightforward. The vermiculite ore was stripped from the mine and hauled in trucks to a mill, where it was separated into various commercial sizes through a screening system. Some of the ore was shipped untouched. Other material was sent to an expansion plant where it was run through ovens at about 2,000 degrees, causing it to expand to 15 times its original size. In 1939, Zonolite merged with another company mining at the bottom of the hill that eventually became known as the Zonolite Company. In 1963, the company was sold to W.R. Grace Company who expanded the operation and increased production. Through the '60s, '70s, and '80s, millions of tons of vermiculite ore was shipped by rail to W.R. Grace plants and other companies in 30 states and six foreign countries. At one time, 80 percent of the world's vermiculite came from Libby, Montana. Piles of vermiculite waste materials remain near the mine and processing facilities in and around Libby, Montana. Vermiculite materials were also used by local citizens as fill, covers on ball parks, gardens, and home insulation. This toxic asbestos has been known to have detrimental affects on former workers at the various facilities and may potentially be affecting the health of Libby residents. Due to this potential threat to human health, the EPA requested emergency environmental response support from the Volpe Center.

Asbestos fibers are odorless and tasteless, and differ in length and structure and chemical composition. Fibers are microscopic and nearly indestructible. They do not evaporate, burn or dry out from heat, or erode in water. Toxicity of different types of asbestos varies, but exposure to any one of them can be fatal. Tremolite, the form found at Libby, is considered by many to be the most toxic.

The human health risks associated with asbestos fibers released in the environment are:

- Malignant mesothelioma, a cancer of the pleural or peritoneal cavity. In early stages of the disease, cancer is found in the lining of the chest cavity near the lung and heart or in the diaphragm. Mesothelioma may spread to tissue surrounding the lungs or other organs. Virtually all mesothelioma cases are attributable to asbestos exposure.

- Asbestosis, the scarring of the tissue of the lung itself from inhalation of fibers. It ranges in severity from mild impairment, to disabling and eventually fatal.

Asbestos and smoking both cause lung cancer, but a history of smoking combined with occupational exposure to asbestos creates a much higher risk of developing the disease.

1.2 PROJECT OBJECTIVES

The primary objectives of the Operable Unit 02 removal action are to:

1) Demolish structures contaminated by asbestos and remove, to the extent possible, all contaminated debris, piles of vermiculite, and soils from the site;

2) Monitor personnel and ensure that airborne asbestos fibers are being properly contained within the removal action area; and

3) Leave the site in suitable condition for reuse with no land use restrictions.

Prior to this removal action, all previous sample collection was performed under the *Sampling and Quality Assurance Project Plan, Revision 1 for Libby, Montana, Environmental Monitoring For Asbestos, Baseline Monitoring for Source Area and Residential Exposure* (EPA 2000). Among objectives of this initial investigation were to rapidly collect pilot-scale information on airborne asbestos levels and data on potential source materials in and around the Libby area. While certain features of this plan remain applicable to the Screening Plant removal action, differing project objectives, more site-specific information, and additional sampling procedures required the development of a new SAP for this effort. The primary objectives of this removal action SAP are to:

1) Confirm that asbestos-contaminated piles and soils have been successfully excavated from Operable Unit 02;

2) Monitor the perimeter boundaries of the site to determine the concentration of asbestos fibers in air during this removal action; and

3) Monitor MARCOR and other personnel, as necessary, to determine exposure to asbestos fibers in air.

1.3 PROJECT ORGANIZATION AND RESPONSIBILITIES

Organization and responsibilities specific to this investigation are listed in this section. For this data collection effort, key management personnel are as follows:

Individual	Role/Responsibility
Paul Peronard	EPA On-Scene Coordinator
Chris Weis	EPA Regional Toxicologist/Scientific Support Coordinator
Jude Hobza	USACOE On-Site Construction Representative for EPA
John McGuiggin	Volpe Center Project Manager
Owen Clark	MARCOR Project Manager
Tim Wall	CDM Federal Task Order Manager
Bob Alexander	CDM Federal Field Team Leader (soil and air)
Mike Hutchinson	CDM Inc. Field Team Member (soil)
Brian Stewart	PES Field Team Leader (air)
Greg Parana	PES Field Team Member (air)
George DeLullo	CDM Federal Quality Assurance Coordinator

2.0 SITE BACKGROUND

It is estimated that tons of asbestos were released into the air during more than six decades of vermiculite mining at the Zonolite Mountain mine, three miles east of Libby, Montana. The W.R. Grace Company, which owned the mine for 30 years, closed it in 1990 and sold its properties four years later.

The former Screening Plant Area, designated by EPA as Operable Unit 02, was utilized by W.R. Grace Company to sort mined vermiculite by grade. The Screening Plant Area, also referred to as the "Railroad Loading Station" or "Raintree Nursery" in previous investigations, is approximately 21 acres (according to county records) in size. Located in the northeast side of the Kootenai River, the site is approximately 4.5 miles northeast of Libby. Vermiculite was transported to the Screening Plant by truck from the mine, located several miles up nearby Rainy Creek Road, and sorted and stored in two sheds at the facility. The vermiculite was then loaded onto a conveyor system and transported across the Kootenai River to the southeast bank for loading onto railroad cars for transportation and distribution to various expansion plants, including the Export Plant in Libby (designated by EPA as Operable Unit 01). The site was most recently operated as a plant nursery, selling nursery-related products and storage for nursery and related supplies, as well as providing leased storage space for recreational vehicles, boats, automobiles, and other items.

Existing structures at the site consist of an office/dwelling and attached solarium, five or six commercial green houses, a shade house, a large shed former used for ore processing, a smaller open-front shed, several auxiliary buildings, and a mobile home. Additional improvements include an above-ground fuel storage tank, an above-ground water storage tank, a large area of asphalt and concrete paving, numerous ornamental plantings, and other landscaping improvements. Piles of waste vermiculite ore containing asbestos remain at the site. Site structures are also contaminated with asbestos as a result of processing activities at the site.

2.1 ENVIRONMENTAL SETTING

Mean annual precipitation in Libby is 19.4 inches, with 37 percent of it occurring in the months of November through January, and 18 percent falling in the months of May and June. The month having the highest average precipitation is January, with 2.42 inches. Temperature in Libby ranges from an average of 22.4 degrees Fahrenheit (° F) in January to an average of 67° F in July. Average annual precipitation at the W.R. Grace mine site is estimated at 30 inches per year (USDA 1977), and the temperature would be expected to average 3 to 5 degrees cooler than at Libby. Climatological data was obtained from the Libby 1 N.E. Ranger station.

Soils in the area have been assigned a Hydrologic Soil Classification of "B" by the Soil Conservation Service. The drainage basin in the vicinity of the mine site is estimated to have >75% ground cover of mature forest in good condition, with moderate slopes. Antecedent moisture is considered to be average. A "Curve Number" of 60 is estimated for both the Rainy Creek and Fleetwood Drainage Basins.

The geology of the site consists of the late Precambrian Belt Group consisting of fine-grained clastic and carbonate rocks which have undergone various degrees of metamorphism, and are covered with glacial outwash and till. The tailings impoundment is located on an intrusive rock body called the Rainy Creek stock, of which Vermiculite Mountain and W.R. Grace's mining area is a part. Depths to bedrock range from less than 2 feet to about 25 feet on the valley walls and from 20 to 45 feet on the valley floor.

Vegetation in the region consists of grasses, coniferous shrubs, and a mixture of deciduous (primarily cottonwood, alder, and aspen) and evergreen trees (cedar, larch, Douglas fir, ponderosa and lodgepole pine, and spruce). Active logging is taking place within the drainage basin of the mine site, both on the mine property and on adjacent Forest Service land.

2.2 PREVIOUS INVESTIGATIONS

As part of initial emergency response action activities conducted in December 1999, 85 soil samples were collected from 71 stations from the primary Screening Plant area and the horse pasture area south of Rainy Creek. Most of the soil samples were surface grab samples collected from a depth of 0 to 2 inches, with co-located subsurface soil samples from a depth of 2 to 12 inches collected for about 20 percent of the surface samples. Depending on the size of the waste pile, composite waste pile samples were collected at varying depths, from a few inches up to 2 feet in depth. Results ranged from non-detect to approximately 4 percent tremolite asbestos.

Additional soil sampling was conducted in the Screening Plant area in March 2000 and April 2000. In March 2000, 17 soil samples were collected from a wooded area north of the green houses and 44 samples were collected from an area referred to as "Government Lot 4" south of the horse pasture. In April 2000, ____samples were collected from a triangular shaped area located between the horse pasture and Highway 37. Results ranged from non-detect to approximately 8 percent tremolite asbestos.

Air sampling technicians, under contract with the EPA's Emergency Response Team , performed limited air sampling at the former Screening Plant. This ambient air sampling was conducted over a two-day period in December of 1999. Ambient air samples were collected within several of the buildings, including the tunnels, at the former screening facility. In addition to the indoor sampling, ambient air samples were also collected at several locations on the former Screening Plant over the past several months.

3.0 DATA QUALITY OBJECTIVES

To ensure that data of sufficient quality and quantity are collected to meet project objectives, the data quality objective (DQO) process (EPA 1993) was utilized to develop DQOs for the soil and air sampling tasks. The DQO process is a series of steps based on the scientific method that are designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate for the intended purpose. The DQO Process consists of the following seven steps:

Step 1:	State the Problem;
Step 2:	Identify the Decision;
Step 3:	Identify Inputs to the Decision;
Step 4:	Define the Study Boundaries;
Step 5:	Develop a Decision Rule;
Step 6:	Specify Limits on Decision Errors; and
Step 7:	Optimize the Design for Obtaining Data.

During the first six steps of the process, the planning team develops decision performance criteria (i.e., DQOs) that will be used to develop the data collection design. The final step of the process involves developing data collection design based on DQOs.

3.1 SOIL

3.1.1 Problem Statement

Soil sampling efforts conducted in late 1999 and early 2000 found asbestos-contaminated soils at Operable Unit 02 as a result of vermiculite ore processing activities. This sampling plan has been developed at ~~the request of the Volpe Center~~ to determine if excavation activities have successfully removed contaminated soil materials from Operable Unit 02. Additionally, pre-excavation sampling may occur to better define the limit and extent of soil and waste removal.

3.1.2 Identify the Decision

Soil data collected during this removal action will be utilized for the following purposes:

1) Confirm that excavation activities have successfully removed asbestos-contaminated soil and waste materials from Operable Unit 02; and

2) Determine the limit and extent of soil and waste materials to be removed, as necessary, either prior to or after excavation has taken place.

Specifically, the data will be used to answer the following questions:

1) Have asbestos-containing waste materials and contaminated soils containing greater than 1 percent asbestos been successfully removed within a specified area (typically, a 100 foot by 100 foot grid)?

2) If the specified area (typically, a 100 foot by 100 foot grid) where removal has occurred still contains asbestos-containing waste materials and contaminated soils containing greater than 1 percent asbestos, what is the limit and extent of excavation needed to reduce asbestos concentrations to less than 1 percent?

3) Is any additional sampling necessary to define the limit and extent of excavation required for this removal action?

3.1.3 Inputs to the Decision

The purpose of this step is to identify the information that needs to be obtained and the measurements that need to be taken to resolve the decision statements. According to the National Emission Standards for Hazardous Air Pollutants (NESHAP) regulations (EPA 1990), a friable asbestos-containing material is defined as "any material containing more than 1 percent asbestos as determined using Polarized Light Microscopy (PLM), that, when dry, can be crumbled, pulverized or reduced to powder by hand pressure." Accordingly, EPA has selected 1 percent asbestos as the action level for soil and waste materials for this removal action.

In order to properly manage and document the success of removal activities, the site will be overlayed with a grid of squares 100 feet by 100 feet. Each grid square will be identified with a unique code (e.g., A3, B6, M7, etc.) and, after initial excavation, composite sampling (usually five subsamples) will be employed to determine the asbestos concentration within each grid square. Grab or composite sampling may also be employed to define the limit and extent of soil material containing greater than 1 percent asbestos prior to excavation or within a grid square after initial excavation has been completed.

All soil samples will be analyzed using National Institute of Occupational Safety and Health (NIOSH) Method 9002 (see Appendix C) for Asbestos (bulk) by PLM. This method is useful for the qualitative identification of asbestos and the semi-quantitative determination of asbestos content of bulk samples. The method measures percent asbestos as perceived by the analyst in comparison to standard area projections, photos, and drawings, or trained experience.

3.1.4 Boundaries of the Removal Action

This step defines the spatial and temporal bounds of the removal action. The horizontal boundaries of the removal action are the Kootenai River to the north and west of the site, Highway 37 to the east, and the southernmost property line of the area referred to as "Government Lot 4". The vertical spatial boundary is from the ground surface to the base contaminated soil or asbestos-containing waste materials. At present, the extent of contamination below ground surface is not known. The removal action focuses on currently known conditions at the site, but historical information may be used to support removal decisions. Therefore, temporal boundaries include the time frame from when the mine was first discovered in the 1880's, and the beginning of vermiculite mining operations in the 1920's, to the time of this removal action in 2000. The decision makers need to know the soil and waste asbestos concentrations to make decisions during this removal. Constraints that could potentially interfere with data collection are denied or ungranted access, physically inaccessible sampling locations, or concerns regarding health and safety.

3.1.5 Decision Rule

The purpose of this step is to define the parameter of interest, specify the action level, and integrate previous DQO outputs into a single statement that describes a logical basis for choosing among removal actions. The parameter of interest is the concentration of asbestos in soil and waste materials. While the primary form of asbestos expected to be encountered is tremolite, the combined concentration of all forms of asbestos will be used for decision making. As discussed previously, the action level is 1 percent asbestos.

3.1.6 Specify Tolerable Limits on Decision Errors

The purpose of this step is to specify the null hypothesis and the decision maker's acceptable limits on decision errors. The null hypothesis for this removal action is that the true concentration of asbestos in soil and waste materials is less than 1 percent. Therefore, the alternative hypothesis is that the true asbestos concentration in soil and waste materials is greater than 1 percent.

Decision makers are interested in knowing the true value of the asbestos concentration. Since the PLM method can only estimate the concentration semi-quantitatively, decisions that are based on these data could be in error. There are several reasons why the decision maker may not know the true asbestos concentration:

1) There may be a high degree of variability of asbestos concentration within a sample. Although a sample may be thoroughly mixed, only a small portion of the sample is used for the analysis. This could result in either an under- or over-estimate of the actual asbestos concentration.

2) Other fibers with optical properties similar to asbestos minerals may give positive interferences. This could result in an over-estimate of the actual asbestos concentration.

3) The optical properties of asbestos may be obscured by a coating on the fibers. This could result in an under-estimate of the actual asbestos concentration.

4) Fibers finer than the resolving power of the microscope (about 0.3 microns [μm]) will not be detected. This could result in an under-estimate of the actual asbestos concentration.

5) Heat and acid may alter the index of refraction of asbestos and change its color. This could result in an under-estimate of the actual asbestos concentration.

6) There may be a high degree of variability of asbestos concentration within the soil and waste materials in-situ. This could result in either an under- or over-estimate of the actual asbestos concentration.

A false positive or "Type I" decision error refers to the type of error made when the null hypothesis is rejected when it is true and a false negative or "Type II" decision error refers to the type of error made when the null hypothesis is accepted when it is false. For this removal action, a Type I decision error would result in deciding that soil or waste materials contained asbestos above the action level ("dirty") when it did not and a Type II decision error would result in deciding that soil or waste materials did not contain asbestos above the action level ("clean") when it did.

The closer the reported concentration is to an action level, the higher the probability that an incorrect decision will be made and, therefore, a "gray region" is normally established which surrounds an action level. For example, a typical "gray region" might be identified as "the action level minus 10%". However, the PLM method is a semi-quantitative method and lacks the necessary precision to establish a suitable "gray region". Therefore, to allow EPA to exercise professional judgement and limit Type II errors, a tolerable decision limit of ±100% of the action level has been established.

In summary, soil and waste materials will be excavated and removed when the asbestos concentration is greater than or equal to 1 percent. Since one objective of this removal action is to leave the site in suitable condition for reuse with no land use restrictions, EPA may elect to remove soil and waste materials reported to contain less than 1 percent asbestos, based on visual evidence, historical information, professional judgement, or other criteria. Conversely, EPA may elect to leave materials in place that contain greater than 1 percent asbestos due to the presence of physical barriers, to prevent excessive environmental damage, based on professional judgement, or other criteria.

3.1.7 Optimize the Decision for Obtaining Data

The purpose of this step is to identify the most resource-effective sampling design that generates data that satisfy the DQOs in the previous steps. The sampling plan described in this SAP are consistent with the DQOs and project objectives for the removal action. However, if during the period of sample collection and/or evaluation, it becomes apparent that the quantity and/or distribution of samples is not sufficient for obtaining the data required to properly characterize soil and waste materials for this removal action, the number, distribution, or methods may be modified to reflect the specific needs of the project. Any changes to this SAP will be approved by EPA and the Volpe Center prior to implementation. In addition, any deviations to this SAP will be noted in the field logbook and/or subsequent data summary reports.

3.2 AIR

3.2.1 Problem Statement

The asbestos removal action being performed at the Screening Plant could potentially produce airborne asbestos concentrations within, and surrounding, the regulated removal action area. These airborne asbestos fibers could present a hazard to those workers within the regulated removal action area and to anyone located in areas surrounding the regulated removal action area. In addition, the release of airborne asbestos fibers during the removal action could potentially further contaminate the regulated removal action area itself and any adjacent properties.

3.2.2 Identify the Decision

Air monitoring data collected during the asbestos removal action will be utilized for the following purposes:

1) Ambient perimeter air samples will be collected prior to the commencement of the removal action to determine background airborne asbestos fiber levels.

2) Ambient perimeter air samples will be collected during daily work activities to ensure that airborne asbestos fibers are not being released during the removal action.

3) Air samples will be collected on a daily basis within the decontamination unit clean room and at the exhaust port of any negative air machines. These ambient air samples shall be used to document that clean rooms are actually clean and that the negative air machines are not exhausting asbestos fibers.

4) At the conclusion of the removal action for the entire project site, final clearance air samples will be collected at the project site. The final clearance air samples shall be compared to the background air samples to ensure that airborne asbestos fiber levels at the completion of the removal action are equal to or lower than the asbestos fiber levels present prior to initiating the removal action.

5) Personal air samples will be collected on the removal action contractor's (MARCOR) workers to document compliance with the Occupational Safety and Health Administration's (OSHA) Asbestos Standard for the Construction Industry (29 CFR Part 1926.1101).

Specifically, the air monitoring data will be collected to answer the following questions:

1) Are airborne asbestos fibers being properly contained within the regulated emergency removal action area?

2) Are the removal action contractor's workers being properly protected from exposure to asbestos fibers in accordance with OSHA Standard 29 CFR Part 1926.1101?

3) Has the performance of the emergency removal action reduced ambient airborne asbestos fiber levels within the regulated emergency removal action area?

4) What type and size of asbestos fibers, if any, are becoming airborne during the emergency removal action?

3.2.3 Inputs to the Decision

To ensure that the air monitoring data are adequate to meet all of the requirements of the decisions listed above, the results of the air sample analysis must include the following information:

- Concentration of asbestos fibers;
- Type of asbestos fibers; and
- Size of asbestos fibers.

Measurement of Asbestos Concentration in Air

There are a number of techniques for measuring asbestos fibers in air, all of which are based on visual identification of structures as asbestos fibers. Most historical human health data and many regulatory limits for asbestos exposure in air are based upon asbestos fiber concentrations measured using phase contrast microscopy (PCM), including OSHA Standard 29 CFR Part 1926.1101. In this method, *fiber material* is defined as having a length of >5 μm and an aspect ratio (length to diameter ratio) of three or more. Results are generally reported in fibers per milliliter of air (f/ml). Turn-around time for results is usually within 24 hours.

More recently, a number of other methods have been developed for quantitative or qualitative measurement of asbestos fibers in air that utilize Transmission Electron Microscopy (TEM), these methods include, but are not limited to, the following:

- The Asbestos Hazard Emergency Response Act (AHERA) TEM Method found in 40 CFR Part 763, *Asbestos-Containing Materials in Schools.*
- International Standards (ISO) Method 10312, *Ambient Air-Determination of asbestos fibres-Direct transfer transmission electron microscopy method.*

These methods are generally more sensitive than PCM, and also allow visualization and quantification of asbestos fibers that are thinner than those visible under PCM analysis.

The use of a combination of the PCM analysis method and both TEM analysis methods will provide the data necessary to meet all of the requirements of the decisions listed above. TEM analysis is more expensive and the analysis takes a longer time to complete than PCM analysis. Therefore, TEM analyses will be used to confirm certain PCM results (usually the worst case samples) and/or when exact mineralogical identification of the asbestos fibers in a sample is desired.

Regulatory Limits

In order to make the decisions required under this section, a number of factors must be considered, including regulatory limits for asbestos in air, background concentrations of asbestos in air collected prior to the emergency removal action, and types of asbestos associated with the known contaminated materials within the regulated emergency removal action area. Table 1, *Summary of Available PCM- and TEM-Based Exposure Levels for Asbestos,* identifies several current asbestos in air exposure levels that may apply to this removal action.

The EPA will determine the most applicable exposure levels, and use these in conjunction with the results of the background perimeter air sampling results, and any other applicable exposure data to define decision exposure limits. However, a default limit of 0.1 fibers per cubic centimeter (f/cc) is assumed for all ambient and personal air monitoring during work activities for this removal action. At a minimum, all personal air monitoring conducted on workers participating in the emergency removal action must meet the requirements of OSHA Standard 29 CFR Part 1926.1101.

3.2.4 Boundaries of the Study

Spatial Boundaries

The horizontal boundaries to be monitored in this project include the entire regulated emergency response action area. This includes the 16 acre Former Screening Facility (regulated area), the perimeter of the regulated area, any decontamination units on and off the site, and all associated equipment and personnel trailers located adjacent to the regulated area. The horizontal boundaries to be monitored also include individuals working within and in the vicinity of the regulated area. The vertical boundary of interest is the ground surface up to approximately 6 feet, which was determined to include the standard human breathing zone.

Temporal Boundaries

There are several factors that can have an affect on airborne asbestos fiber concentrations. Concentrations of airborne asbestos fibers may vary within and around the regulated area depending on tasks performed, amount of demolition activity, weather, wind direction and velocity, etc. Therefore, both personal and ambient air monitoring will be performed over the duration of the project. In addition, short term exposure limit (STEL) air samples will be collected on removal action contractor employees each time a new task is initiated. This sampling will be performed in accordance with OSHA Standard 29 CFR Part 1926.1101.

3.2.5 Decision Process

Ambient Air Monitoring

As stated earlier, the EPA will be required to determine the most applicable exposure levels for the ambient air monitoring, and use these in conjunction with the results of the background perimeter air sampling results, and any other applicable exposure data to define decision exposure limits. If the agreed-upon exposure limits are not exceeded during the course of the removal action, work can proceed as planned. If the exposure limits are exceeded during the removal action, work will be stopped and dust suppression methods, such as the amount of water used by the removal action contractor, should be reevaluated. Work can continue once the airborne asbestos fiber concentrations have been lowered below the exposure limits agreed upon prior to the project. As a default value, a 0.1 f/cc exposure limit for perimeter sampling during removal activities and for clearance sampling at the end of the project is assumed.

Personal Air Monitoring

The EPA may also be required to determine the most applicable exposure levels for the personal air monitoring during the course of the removal action. At a minimum, all personal air monitoring conducted on workers participating in the emergency removal action must meet the requirements of OSHA Standard 29 CFR Part 1926.1101. Unless notified otherwise, a default exposure limit of 0.1 f/cc is assumed. If these personal exposure limits are not exceeded during the course of the removal action, work can proceed as planned. If these exposure limits are exceeded during the removal action, work should be stopped and dust suppression methods, such as the amount of water used by the removal action contractor, and/or respiratory protection requirements should be reevaluated. Work can continue once the airborne asbestos fiber concentrations have been lowered below acceptable exposure limits. Work can also continue if the removal action contractor institutes revised respiratory protection requirements that ensure the workers are not exposed to the airborne asbestos fiber concentrations. However, a change in respiratory protection should be a last resort. Lowering the airborne fiber concentrations within the regulated area through engineering controls should always be the first option.

3.2.6 Specify Tolerable Limits on Decision Errors

Due to the size of the regulated emergency removal action area, the "worst case" ambient air sample and the "worst case" personal air sample will be used to make risk management decisions during the removal action. Each day the ambient air sample and the personal air sample with the highest concentration of asbestos fibers will be used to determine the airborne fiber levels of the entire regulated area. For this removal action, the null hypothesis is that the true concentration of asbestos fibers in air is below the exposure limit. The alternative hypothesis is that the true concentration of asbestos fibers in air is above the exposure limit.

As discussed earlier in the DQOs for soils, false positive or "Type I" decision error refers to the type of error made when the null hypothesis is rejected when it is true and a false negative or "Type II" decision error refers to the type of error made when the null hypothesis is accepted when it is false. For this removal action, a Type I decision error would result in deciding that an air sample result was above the exposure limit ("dirty") when it was not and a Type II decision error would result in deciding that an air sample result was not above the exposure ("clean") when it actually was.

The closer the reported concentration is to a action level, the higher the probability that an incorrect decision will be made and, therefore, a "gray region" is normally established which surrounds an action level. Air monitoring for this removal action is primarily being performed to determine if excessive levels of asbestos fibers migrating off-site and for health and safety purposes. Since soil and waste material removal actions will not be determined based on the results of air sampling, a "gray region" was not established. However, because human health and safety are involved, a tolerable decision limit of ±50% is prudent. This decision limit would allow, for example, the implementation of additional control measures (e.g., additional dust suppression measures) before exposure limits are exceeded, based on professional judgement, at one half of the exposure limit.

3.2.7 Optimize the Decision for Obtaining Data

Both ambient air monitoring and personal air monitoring will be conducted daily during the emergency action. Risk management decisions will be reevaluated daily by the EPA. If it becomes apparent over the course of the removal action that the quantity or distribution of the air samples may not be sufficient for obtaining the data required to characterize the airborne asbestos fiber concentrations within, and adjacent to, the regulated removal action area, the number and location of air samples may be modified to meet the needs of the project. The decision to modify the sampling strategy will be determined by the EPA.

4.0 SAMPLING METHODS AND PROCEDURES

CDM Federal was tasked by the Volpe Center to provide confirmation sampling of soil, and perimeter and personal air for asbestos for the Operable Unit 02 removal action. This section describes the procedures that will be followed for sample collection, handling, analysis, and documentation. Sample management diagrams for soil and air samples are shown on Figures 2 and 3, respectively.

4.1 SOIL

4.1.1 Selecting Soil Sampling Locations

Confirmation sampling will be conducted within excavation grids 100 feet x 100 feet in size to determine if contaminated soil materials have been successfully excavated from each grid. Excavation grids have been designated on the construction drawings. Each grid square will be identified with a unique code (e.g., A3, B6, M7, etc.). Samples from each grid will consist of five subsamples to form a composite sample. Typically, one subsample will be collected from the center of the grid with the remaining four subsamples collected approximately 25 feet from the center subsample in a cross-shaped pattern. The directional orientation of the cross will be randomized using a game spinner, a table of random compass bearings, or other appropriate method. Adjustments to this pattern may be made based on field conditions (e.g., to account for the presence of trees or other obstructions, the presence of building foundations, concrete pads, surface water features, or unexcavated areas). Approximate subsample locations shall be recorded on the field sample data sheets. Examples of the field sheets are provided in Appendix A.

Other soil samples may be collected to further define the limit and extent of contaminated soil material prior to the removal action or within a grid square after initial excavation has been completed. These may be grab or composite samples, depending on the type of information required. The type and location of these samples will be documented on the field log sheets.

4.1.2 Sample Identification

Each soil sample will be identified with two unique codes. The first code is an index identification code taken from a list of unique codes prepared by ISSI Consulting Group, Inc. (ISSI), a contractor to EPA. This coding system is designed to prevent accidental duplication of sample identification numbers and ensures that all samples have a unique identification number assigned to them. During previous sampling events, these codes started from 1-00001 and the last five numbers were sequentially numbered so that thousands of unique codes would be available, if necessary. To ensure that the laboratory is "blind" and does not receive certain specific information about a sample, only the index identification code, along with sample date and time, will be used to label sample containers.

The second sample code is a field identification code used by CDM Federal to give each soil sample a unique identity that will allow for the tracking and retrieval of information concerning each sample. The structure of the coding system will be similar to the coding system used in previous sampling events. Each surface soil sample will be identified, at a minimum, by a site identifier, a location identifier, a media identifier, a station identifier, and the depth interval of the sample collected. For example, LIB-FSPA-SO-001-0-2 would indicate that a sample was collected by CDM Federal as part of the Libby asbestos investigation (LIB-), that it was collected from the Former Screening Plant Area (FSPA-), that is was a soil sample (SO-), that it was from sampling station 001 (001-), and that it was collected from the 0- to 2-inch depth interval (0-2). Modifications to this basic coding system may be necessary to identify the grid location or number, whether it was a grab or composite sample, sample date, a duplicate sample, etc. The actual coding system used will be clearly described in the field logbook.

4.1.3 Collecting Soil Samples

All soil samples will be collected in accordance with CDM Federal Standard Operating Procedure (SOP) 1-3, Surface Soil Sampling, with modifications (Appendix B). The following modifications to SOP 1-3 have been reviewed and approved:

Section 2.2, Discussion - Unless otherwise directed by EPA or the Volpe Center, sample depth will be 0 to 2 inches.

Section 4.0, Required Equipment - Neither ice bags nor blue ice will be used. However, the samples will be kept as cool as possible at all times. Powder-free nitrile gloves will be used for sample collection. No pans, trays, or bowls will be necessary, since samples will be placed directly into zip-top bags. Since the sampling is for asbestos, rather than metals or organics, the use of stainless steel or Teflon-lined sampling instruments is not necessary.

Section 5.2.3, Method for Collecting Samples for Nonvolatile Organic or Inorganic Compound Analysis - Zip-top bags will be used as the sample containers. Sampling information will be written directly on the bags using a permanent marker. Sampling instruments will not necessarily be constructed of stainless steel or be Teflon lined. Trays and bowls will not be used, as samples will be placed directly into zip-top bags. Homogenization will be performed by manipulating the sampled materials inside the zip-top bag. All samples will be double bagged.

Initial sample homogenization will be accomplished in the field by gently kneading the contents of the sealed bag to break up any remaining soil clumps, then mixing by turning the bag slowly end-over-end a minimum of ten times. All other sample preparation, such as sample drying, shall be completed by the EMSL on-site or off-site laboratory.

4.1.4 Sample Documentation

Sampling activities during this removal action will be documented in a field logbook (and on field sample data sheets, see Appendix A) to be maintained by the field team in accordance with CDM Federal SOP 4-1, Field Logbook Content and Control (Appendix B). The Field Team Leader (FTL) is responsible for maintenance and document control of the field logbook. In addition, all sample locations will be surveyed using standard surveying equipment and/or a Global Positioning System (GPS) unit.

4.1.5 Sample Custody, Packaging, and Shipping

Sample custody includes the classifying, identifying, labeling, packaging, and transporting of samples collected during this investigation. Procedures for handling samples collected during this investigation will follow CDM Federal SOPs 1-2, 2-5, and 4-5 (Appendix B) as described below.

Sample classification is necessary to ensure the protection of personnel involved in the shipment of samples, and to maintain the integrity of each sample. Samples obtained at uncontrolled hazardous waste sites are classified as either environmental or hazardous samples. All samples collected during this investigation will be classified as environmental.

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the off-site laboratory, chain-of-custody (COC) records will be used. The COC record is employed as physical evidence of sample custody and control, and provides the means to identify, track, and monitor each individual sample from the point of collection through disposal. COC procedures will follow the requirements set forth in CDM Federal SOP 1-2, Sample Custody, with modifications (Appendix B). The following modifications to SOP 1-2 have been reviewed and approved:

<u>Section 5.1, Chain-of-Custody Record</u> - Electronic chain-of-custody forms will be generated using Microsoft Access in the Libby database system developed by ISSI for EPA or as specified by the Volpe Center.

<u>Section 5.2, Sample Labels and Tags</u> - Samples will be identified by writing sample information directly on the one-gallon zip-top bags using permanent markers. All samples will be double-bagged.

Samples collected during this investigation that require analysis at on- or off-site laboratories will be packaged and shipped in accordance with CDM Federal SOP 2-5, Packaging and Shipping of Environmental Samples, with modification (Appendix B). The following modifications to SOP 2-5 have been reviewed and approved:

<u>Section 4.0, Required Equipment</u> - No vermiculite or other absorbent material, bubble wrap, or ice will be used.

<u>Section 5.0, Procedures</u> - No vermiculite or other absorbent material will be used to pack the samples. No ice will be used. Maintenance of temperatures less than or equal to 4 derees Celsius was determined by EPA not to be critical, however, the samples will be kept as cool as possible during all phases of work.

4.1.6 Quality Control Samples

Quality control (QC) data are necessary to determine precision and accuracy of sample collection techniques and to help determine any variability that exists within a sample. For this investigation, soil QC samples will consist of duplicates collected from the sample bag after thorough homogenization. Soil QC samples will be analyzed at a rate of one per ten samples (i.e., 10 percent) or at the rate specified by the Volpe Center.

4.1.7 Equipment Decontamination

Equipment used to collect, handle, or measure soil samples will be decontaminated in accordance with CDM Federal SOP 4-5, Field Equipment Decontamination at Nonradioactive Sites, with modification (Appendix B). The following modifications to SOP 4-5 have been reviewed and approved:

<u>Section 5.0, Procedures</u> - Decontamination water will not be captured. No solvents will be used for decontamination.

<u>Section 5.3, Sampling Equipment Decontamination</u> - ASTM Type II deionized (DI) water will not be used. Rather, locally available DI water will be used. Nitric acid will not be used. Decontamination water will be disposed of on-site.

<u>Section 5.6, Waste Disposal</u> - Decontamination water will be disposed of on-site and will not be packaged, labeled, or stored as investigation-derived waste.

The decontamination procedure for non-disposable equipment will consist of a tap water and alconox wash with brush scrubbing, followed by a tap water rinse, and final DI water rinse. The equipment will then be allowed to air-dry before being wrapped in clean plastic or aluminum foil. All equipment will be decontaminated before coming into contact with any sample. Rinse water will be discharged to the ground at the site. Any deviations from the decontamination procedures will be recorded in the appropriate field logbook.

<u>4.2 AIR</u>

4.2.1 Selecting Sample Locations

Ambient Air-monitoring

Background ambient air samples, ambient air samples collected during the removal action, and final clearance ambient air samples, will be collected at six fixed perimeter monitoring sites. These six sites surround the regulated emergency removal action area. Two sites are located on the northern perimeter of the site, two sites along the southern perimeter, one on the east perimeter, and one on the west perimeter. The locations of these sites were selected to ensure that airborne asbestos fiber concentrations migrating from the regulated removal action could be determined independent of wind direction or work location.

In addition to the perimeter air samples, additional ambient air samples will be collected depending on the day's work activities. These samples will be collected at locations such as the decontamination chambers, negative air machines, contractor trailers, etc. The number and location of these additional ambient air samples will be determined by the air-monitoring consulting firm along with the EPA and the Volpe Center.

Personal Air Monitoring

Personal air samples on the removal action contractor's (MARCOR) workers will be conducted to document compliance with 29 CFR Part 1926.1101. All personal air samples shall be collected and analyzed by EMSL Analytical Inc. (EMSL), in accordance with 29 CFR 1926.1101.

4.2.2 Sample Identification

The air-monitoring consulting firm will use the sampling numbering scheme that was designed for the removal action by ISSI, a subcontractor to the EPA. This scheme consists of the precursor "1R" followed by a dash, and then by a six digit sampling number (1R-000001, 1R-000002, etc.). The sampling numbers are issued and signed out in accordance with ISSI's sample numbering protocols. Sample details will be noted on the air sampling log sheets.

4.2.3 Collecting Samples

Air samples will be collected by drawing air through a cellulose acetate filter (0.45 μm pore size) at a specified flow rate for a specified period of time. The details of the method are provided in EPA SOP 2015. During normal working activities both ambient and personal air samples will be collected at a flow rate of 2.5 liters per minute (l/min) over an 8-hour sampling period. This will result in a total sampling volume of 1200 liters.

Depending on the sampling conditions, work activities, the level of asbestos in the air, and the level of interfering particles in the air, the flow rate and or total sampling time may require modifications. The decision to modify the flow rate or total sampling time will be made by the FTL, in conjunction with the EPA On-Scene Coordinator.

4.2.4 Sample Custody, Documentation, Packaging, and Shipping

Documentation of sample collection, handling, and shipment will include completion COC forms in the field, field forms, and entry of data into a field logbook. Each sample will be properly labeled with a unique sample identifier. A COC form shall accompany every shipment of samples to the analytical laboratory. The purpose of the COC form is to establish the documentation necessary to trace possession from the time of collection to final disposal. Figure 1, Sample Management Flow Diagram for Air Samples, summarizes the sampling, handling, and analyses for each sample type.

The COC will be designed by ISSI. Minimally, the field form will have the following information:

- Project number;
- Sampler's signature;
- Sample volume;
- Sample identification number; and
- Analytical parameters.

The shipping forms or transmittal memo from the EPA will describe:

- Number of samples;
- Sample preservation (if applicable); and
- Date and time of sample shipments.

The laboratory will enter the following information upon receipt:

- Name of person receiving the sample;
- Date of sample receipt; and
- Sample condition.

All corrections to the COC record will be initialed and dated by the person making the corrections. Each COC will include signatures of the appropriate individuals indicated on the form. The originals will accompany the samples to the laboratory, and copies documenting each custody change will be recorded and kept on file. Copies of all COCs will provided to the Volpe Center.

Chain-of-custody will be maintained until final disposition of the samples by the laboratory and acceptance of the analytical results by the EPA. One copy of the COC will be kept by field personnel.

The microscopist will include the following information on the field form:

- Date;
- Microscopist's name;
- Sample identification;
- Mineral type; and
- Structure counts.

All required paperwork, including sample labels, COC forms, custody seals, and shipping forms will be fully completed in ink (or printed from a computer) prior to shipping of the samples to the laboratory. Shipping from sample storage to the off-site laboratory will be by overnight delivery and delivery to the on-site laboratory will be by field personnel.

Upon receipt, the samples will be given to the laboratory sample custodian. The boxes will be opened and the contents inspected. Chain-of-custody forms will be reviewed for completeness, and samples will be logged and assigned a unique laboratory sample number. Any discrepancies or abnormalities in samples will be noted.

4.2.5 Quality Control Samples

Quality control methods will include both a field and laboratory component. Field personnel will prepare two types of QC samples: replicates and blanks.

Replicates

For air samples, replicates are defined as separate samples that are collected using separate air pumps and filters. These air samples are collected side-by-side at a location and are sampled for the same amount of time. Air pumps are set at the same airflow rates so that adequate and like air volumes are passed through each filter.

Blanks

Field personnel will prepare blank samples for air by labeling unused filter cassettes and submitting them for analysis.

The laboratory staff will have the responsibility of processing all samples submitted according to the specific protocols for sample custody, analysis, reporting, and associated laboratory Quality Assurance/Quality Control.

Quality assurance programs for analytical chemistry typically include blanks, blind standards, and spikes. Performance evaluation using blind standard and spikes are not available for asbestos analysis. The laboratory quality assurance program will consist of blanks and replicate analysis. Blank samples will be used to assess contamination in the filter medium. A subset of TEM grids (10-20%) will be sent either back to the primary laboratory for re-analysis (blind) or to additional laboratories for replicate analysis, as directed by the Volpe Center. The TEM grids for replicate analysis will be shipped under COC to appropriate laboratories for analysis.

4.2.6 Equipment Decontamination

This project requires the decontamination of all air sampling equipment (e.g., pumps, cassette holders, etc.) that is used within the regulated emergency response action area prior to it being removed from that area. All air sampling pumps, tubing, sampling stands, and rotometers will be decontaminated at the end of each sampling day. All equipment will be wiped down with DI water, and dried with clean disposable wipes or rags. These rags will be disposed of as asbestos-contaminated waste. Once the equipment has been decontaminated, it will be stored outside of the regulated area during non-sampling hours.

4.3 HEALTH AND SAFETY

All sampling shall be performed in accordance with all applicable EPA, OSHA, corporate, and site health and safety requirements. More specifically, when working within the regulated emergency removal action area, CDM Federal and its subcontractors will follow all requirements of the removal action contractor's (MARCOR) site specific health and safety plan.

5.0 LABORATORY ANALYTICAL METHODS

Twenty-four hour laboratory services will be provided in an on-site laboratory, which will analyze the soil samples, personal air, and certain perimeter air samples. An off-site laboratory will provide analytical support for a portion of the air samples collected.

The most appropriate analytical methods for each environmental medium depend on the type and level of asbestos contamination and on the detection levels needed to assess hazard and/or nature and extent of contamination. Table 2, *Summary of Analytical Methods*, identifies the analytical methods which will be utilized during the emergency removal action. Copies of these analytical methods are provided in Appendix C. The table also shows some of the requirements and characteristics of each method. All of these analytical methods were determined by the EPA.

The laboratory used for all sample analysis must be accredited under the Laboratory Accreditation Program as sponsored by the American Industrial Hygiene Association (AIHA). The laboratory must also actively participate in the NIOSH *Proficiency Analytical Testing Program for Laboratory Quality Control* for asbestos. Lastly, the laboratory must be fully accredited for TEM and PLM analysis under the National Voluntary Laboratory Accreditation Program as sponsored by the National Institute of Standards and Technology (NIST).

6.0 REFERENCES

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AHERA.

EMSL. 1985.

EPA. 1990. Asbestos/NESHAP Regulated Asbestos Containing Materials Guidance. EPA 340/1-90-018. December.

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EPA. 1999. Sampling and Analysis Plan for the Preliminary Investigation of Environmental Asbestos. November 24.

EPA. 2000. Sampling and Quality Assurance Project Plan, Revision 1 for Libby, Montana, Environmental Monitoring For Asbestos, Baseline Monitoring for Source Area and Residential Exposure.

IRIS. 1999. National Research Council/National Academy of Sciences: Asbestiform Fibers - Nonoccupational Health Risks.

OSHA. 1998. Occupational Exposure to Asbestos. 29 CFR Part 1919.1001. Specifically Regulated Chemicals - Subpart Z - Toxic and Hazardous Substances.

OSHA. 1998. Occupational Exposure to Asbestos. 29 CFR Part 1926. Safety and Health Regulations for Construction.

USDA. 1977.

FIGURES

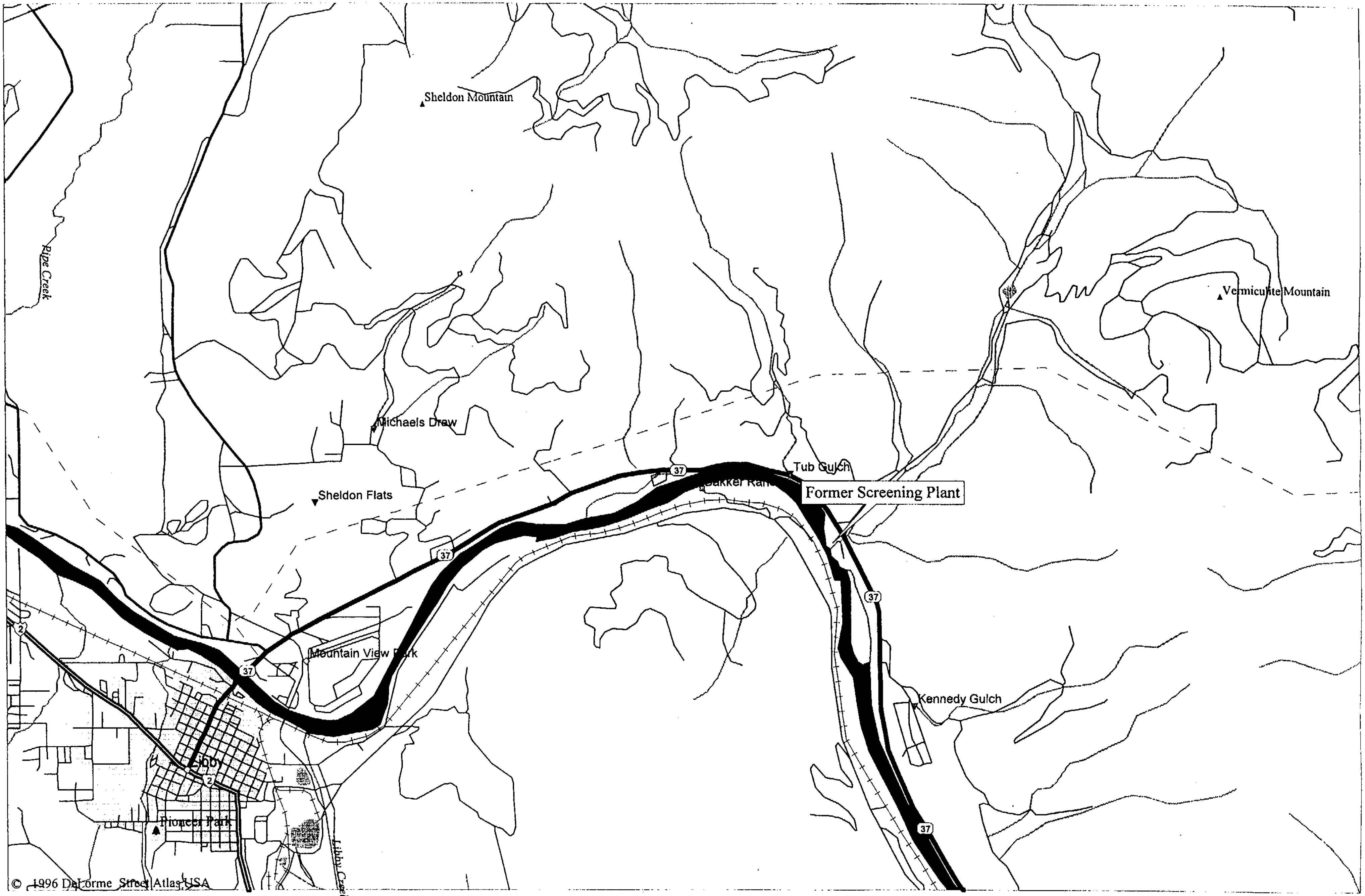


Figure 1. Site Map Showing Area of Study

OPERABLE UNIT 02
FORMER SCREENING PLANT AREA
NEAR LIBBY, MONTANA

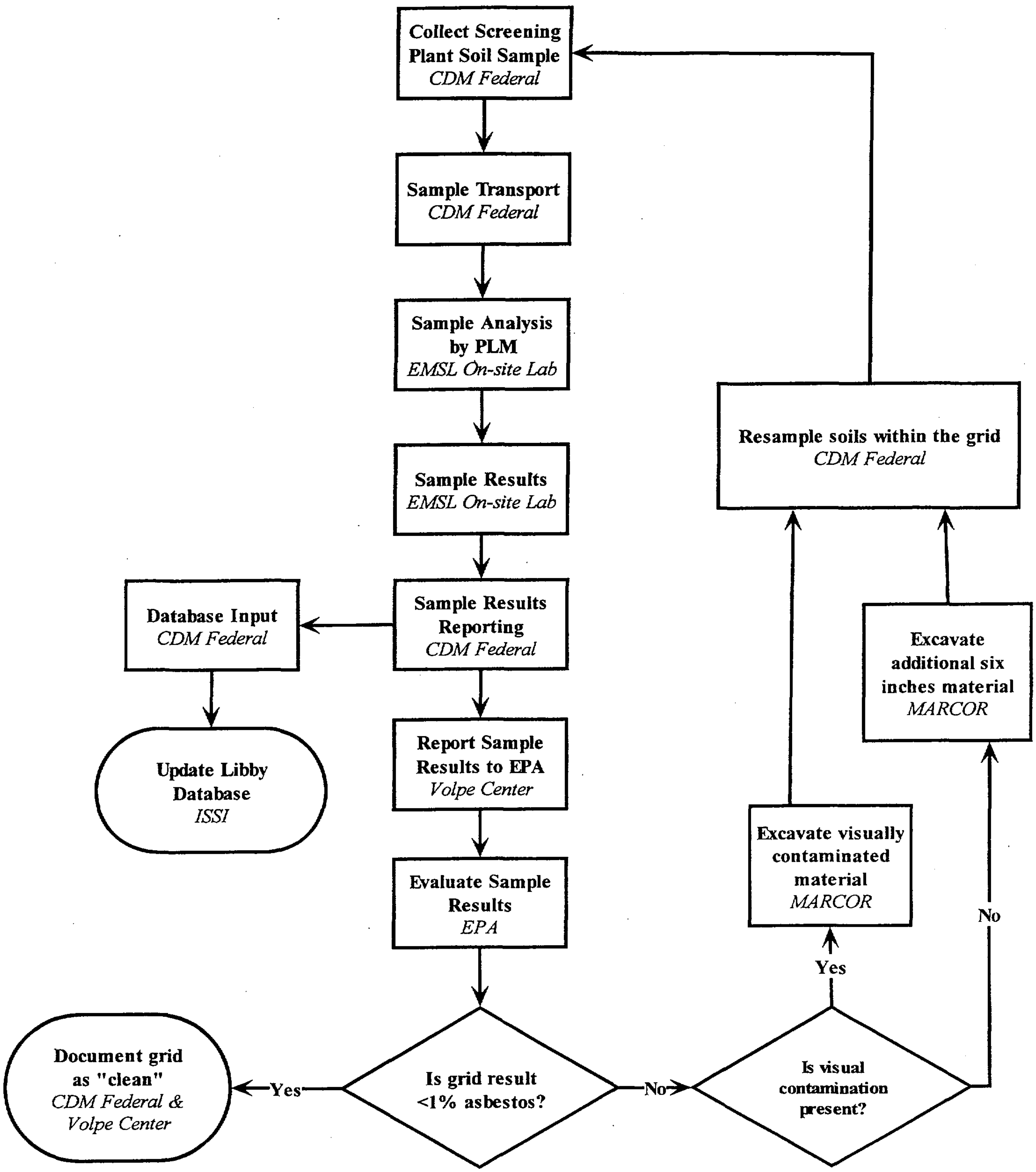


Figure 2. Sample Management Flow Diagram for Soils

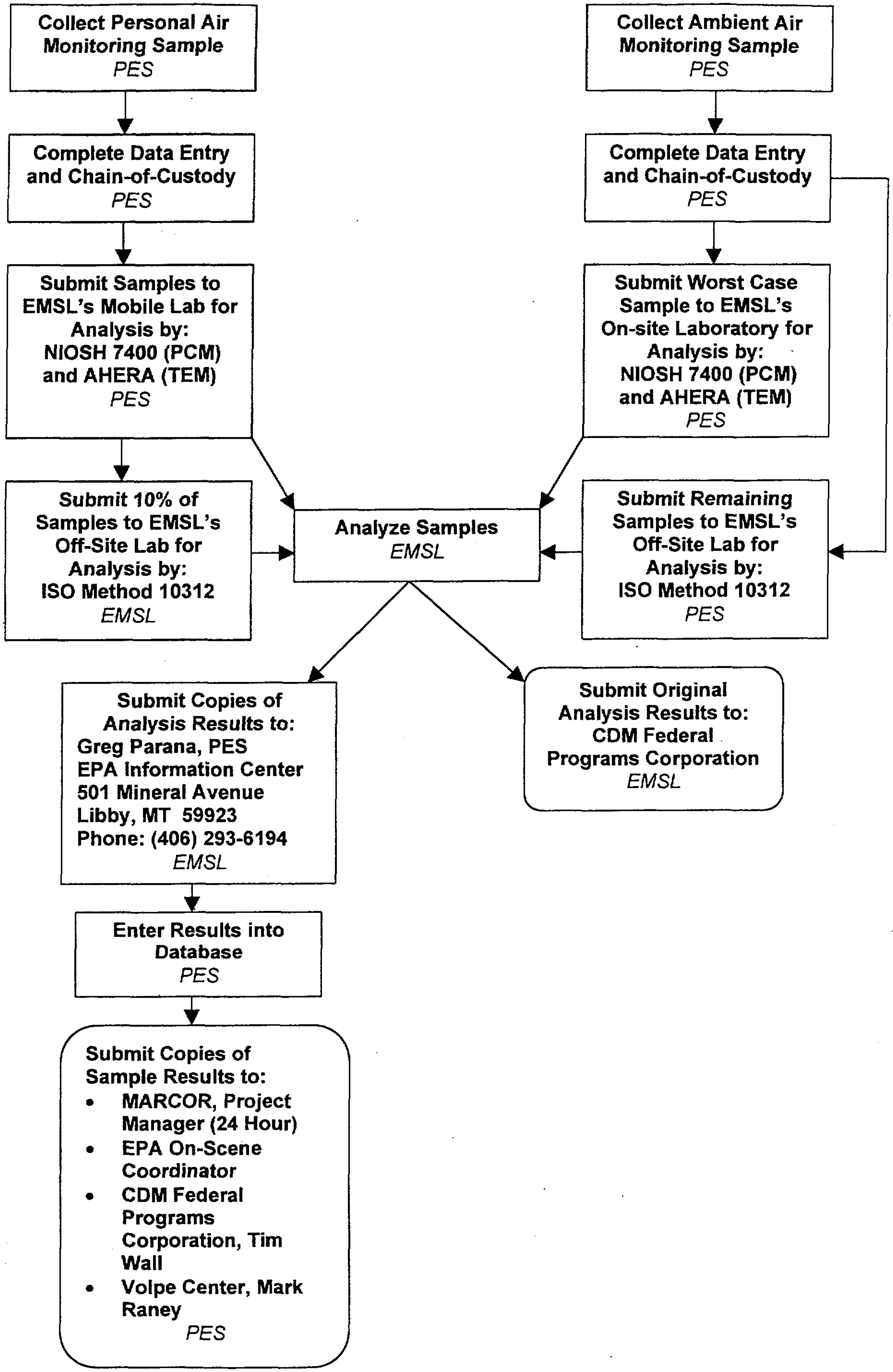


Figure 3. Sample Management Flow Diagram for Air Samples

TABLES

Table 1
Summary of Available PCM- and TEM-Based Exposure Levels for Asbestos

Agency	Description	Required Analysis	Nominal Value	Reference
ACGIH	TLV-TWA	PCM	0.1 f/cc (0.1 f/ml)	ACGIH 1998
NIOSH	REL 100-minute TWA in a 400-liter sample (all forms)	PCM	0.1 f/cc (0.1 f/ml)	NIOSH 1999
OSHA	PEL (TWA) all forms	PCM	0.1 f/cc (0.1 f/ml)	OSHA 1998 29 CFR 1919.1001
OSHA	PEL (ceiling) 30 minute average (all forms)	PCM	1.0 f/cc (0.1 f/ml)	OSHA 1998 29 CFR 1926.1101
EPA (AHERA)	Level to determine the completion of a response action in Schools	TEM	70 structures per square millimeter (s/mm^2)	EPA 1987 40 CFR 763
EPA (IRIS)	Inhalation unit risk	PCM	0.23 f/ml	IRIS 1999
EPA (EMSL)	Measure of Work Site Cleanliness	PCM	Less than or equal to 0.01 f/cc (0.1 f/ml)	EMSL 1985
EPA (OW)	MCL (f>10 μm in length) all forms	PCM	7 million fibers per liter (MFL) (7000 f/ml)	EPA 1998

Table 2
Summary of Analytical Methods

Matrix	Analysis	Sample Media	Holding	Sample Preparation	Analytical Method
Air	PCM	Three-piece, 25 mm cassette, mixed cellulose ester filter, 0.45 micron pore size	6 months	Select area of filter for prep. Cut filter. Place filter on slide and insert into 'hot block'. Clean filter with acetone vapor. Affix filter to slide with triacetone and place cover slide. Affix coverslide with nail polish. Outline filter piece with glass marking pen.	NIOSH Method 7400 (see Appendix C)
Air	TEM	Three-piece, 25 mm cassette, mixed cellulose ester filter, 0.45 micron pore size	6 months	Select area of filter for prep. Cut filter. Collapse the cut portion of filter using dimethylformamide and acetic acid solution onto slide. Plasma etch the filter portion. Coat the microscope slide with carbon. Wash the slide using the Jaffe Washer.	EPA AHERA Method (see Appendix C)
Air	TEM	Three-piece, 25 mm cassette, mixed cellulose ester filter, 0.45 micron pore size	6 months	Select area of filter for prep. Cut filter. Collapse the cut portion of filter using dimethylformamide and acetic acid solution onto slide. Plasma etch the filter portion. Coat the microscope slide with carbon. Wash the slide using the Jaffe Washer.	ISO 10312 (see Appendix C)
Soil	PLM	None	6 months	Use forceps to sample at several places from the bulk material. Forcep samples are immersed in a refractive index liquid on a microscope slide, teased apart, covered with a cover glass, and observed with the polarized light microscope.	NIOSH 9002 (see Appendix C)

Table 3
Sampling Supply and Equipment Checklist
Libby Asbestos Project

Note: This supply and equipment list should be used in addition to the list found in the specific SOPs.

General

SAP
SOPs
HSP
pens
permanent markers
field book
pin flags
digital camera
garbage bags
100-foot tape measure
GPS unit
cellular phone
file box
color pencils
express shipping labels
field forms
tool kit

Equipment Decontamination/Personal Protective Equipment

rubber overboots
tyvek coveralls
liquid soap
disposable gloves
respirators w/ HEPA-type cartridges
duct tape
paper towels
1-gal zip-top bags
respirator cleaning kit
5-gal water-boy
paper towels
safety glasses
eye wash kit
first aid kit
personal decontamination supplies:
- one tub soapy water
- one tub tap water
- garden sprayer

long-handled brush
aluminum foil

Soil Sampling

bulb planting tool, trowel, or other sampling device
ice pick, pulaski, or spud bar (in case of frozen ground)
site map
wood stakes
300-ft measuring tape
flagging
plastic sheeting

Air Sampling

high-volume sample pumps
low-volume sample pumps
tubing
sample stands
air-flow calibrator
tubing/cassette adaptors
sample labels/tags
shrink-wrap
extension cord
filters (0.45 μm?)
air sampling forms
filter cassettes
step-stool
tyvek
metric ruler
filter paper
81/2 x 11 plastic sheets
masking tape
disposable hand cleaning wipes
tape measure
access agreement
flash light w/batteries
ear plugs
magnifying glass

APPENDIX A

FIELD SAMPLE DATA SHEETS

Sheet No: S-______

LIBBY MONTANA SITE INVESTIGATION
FIELD SAMPLE DATA SHEET FOR

SOIL-LIKE MATERIALS

Address:______________________________
Address or Location ID:______________________________
GPS (if no address available): Northing______________ Easting______________
Owner:______________________________
Land Use Category: Residential School Commercial Mining Other (__________)

Site Visit Date:______________ Sampling Team:______________ Sample Time:______

Data Item	Sample 1	Sample 2	Sample 3
Field ID Number			
Index ID			
Matrix Type (circle)	Yard Soil Garden Soil Driveway Mining Waste	Yard Soil Garden Soil Driveway Mining Waste	Yard Soil Garden Soil Driveway Mining Waste
Category (circle)	FS FD________	FS FD________	FS FD________
Type (circle)	Grab Comp	Grab Comp	Grab Comp
Top Depth (in.)			
Bottom Depth (in.)			
Map location(s) (Indicate on field sketch—over)			
Comments			

Sheet No: A-_____

LIBBY MONTANA SITE INVESTIGATION
FIELD SAMPLE DATA SHEET FOR

AIR

Address or Location ID:_______________________________

GPS (if no address available): Northing_______________ Easting_______________

Owner:_______________________________

Land Use Category: Residential School Commercial Mining Other (____________)

Site Visit Date:____________________ Sampling Team:____________________

Data Item	**Cassette 1**	**Cassette 2**	**Cassette 3**
Field ID Number			
Index ID			
Category (circle)	FS Rep________ Blank	FS Rep________ Blank	FS Rep________ Blank
Matrix Type (circle)	Indoor Outdoor	Indoor Outdoor	Indoor Outdoor
Location Description			
Flow Meter Type			
Flow Meter ID No.			
Pump ID Number			
Start-Date			
Start-Time			
Start-Flow (L/min)			
Start-Counter			
Stop-Date			
Stop-Time			
Stop-Flow (L/min)			
Stop-Counter			
Pump fault?	No Yes	No Yes	No Yes
MET Station onsite?	No Yes	No Yes	No Yes
Field Comments			

APPENDIX B

STANDARD OPERATING PROCEDURES

CDM FEDERAL TECHNICAL STANDARD OPERATING PROCEDURES

SOP 1-2	Sample Custody
SOP 1-3	Surface Soil Sampling
SOP 2-5	Packaging and shipping of Environmental Samples
SOP 4-1	Field Logbook Content and Control
SOP 4-5	Field Equipment Decontamination at Nonradioactive Sites

SAMPLE CUSTODY

Prepared: David O. [illegible] 12/23/92 Signature/Date
Technical Review: [illegible] 1-4-93 Signature/Date
QA Review: Teresa Neli 1/2/92 Signature/Date
Approved: [illegible] 3/11/93 Signature/Date
Issued: Rose Mary Ellersick 4/23/93 Signature/Date

1.0 OBJECTIVE

Due to the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures are followed. All paper work associated with the sample custody procedures will be retained in CDM Federal Programs Corporation (CDM Federal) files unless the client requests that it be transferred to them for use in legal proceedings or at the completion of the contract.

2.0 BACKGROUND

2.1 Definitions

<u>Sample</u> - A material to be analyzed that is contained in single or multiple containers representing a unique sample identification number.

<u>Sample Custody</u> - A sample is under custody if:

1. It is in your possession.
2. It is in your view, after being in your possession.
3. It was in your possession and you locked it up.
4. It is in a designated secure area.

<u>Chain-of-Custody Record</u> - Form used to document the transfer of custody of samples from one individual to another.

<u>Custody Seal</u> - A custody seal is a tape-like seal that is part of the chain-of-custody process and is used to prevent tampering with samples after they have been packed for shipping.
<u>Sample Tag</u> - Tag generated by EPA's Contract Laboratory Program (CLP) for designating a CLP sample number on samples.

3.0 RESPONSIBILITIES

Sampler - The sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

Site Manager - The site manager is responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events.

4.0 REQUIRED EQUIPMENT

- Chain-of-Custody Records (applicable CLP or CDM Federal forms)
- Custody Seals
- Sample Tags

Note: The CLP sample coordinator will supply the appropriate Chain-of-Custody Records, sample tags, and CLP sample numbers.

5.0 PROCEDURES

5.1 Chain-of-Custody Record

This procedure establishes a method for maintaining custody of samples through use of a Chain-of-Custody Record. This procedure will be followed for all samples collected or split samples accepted.

<u>Field Custody</u>

1. Collect only the number of samples needed to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations prior to the actual field work. As few people as possible should handle samples.

2. The field sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

3. Sample labels shall be completed for each sample, using waterproof ink; sample tags shall also be completed for CLP work.

4. The Site Manager determines whether proper custody procedures were followed during the field work and decides if additional samples are required.

Transfer of Custody and Shipment

1. Samples are accompanied by a Chain-of-Custody Record (see Figure 1; Chain-of-Custody Record). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst in the appropriate laboratory.

   - The date/time will be the same for both signatures when custody is transferred directly to another person. When samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures. Common carriers are not required to sign the form.

   - In all cases, it must be readily apparent that the person who received custody is the same person who relinquished custody to the next custodian.

   - If samples are left unattended or a person refuses to sign, this must be documented and explained on the Chain-of-Custody Record.

2. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment.

3. All shipments will be accompanied by the Chain-of-Custody Record identifying its contents. The original record will accompany the shipment, and the copies will be retained by the Site Manager and if applicable, distributed to the appropriate CLP sample coordinators. Freight bills will also be retained by the Site Manager as part of the permanent documentation.

Procedure for Completing CDM Federal Chain-of-Custody Record

Note: Refer to EPA Regional guidelines for completing CLP custody records.

1. Record Project Number.

2. Record Field Team Leader for the project.

3. Record the name and address of the laboratory to which samples are being shipped.

4. Record the record number and total number of records being shipped for the day. ✓

5. Enter the project name/location or code number. ✓

6. Record overnight courier's airbill number. ✓

7. Note sample type (matrix) and reference number. Include reference number if requested on the Chain-of-Custody Record, box 9.

8. Record sample identification number.

9. Enter the reference number from box #7. Record equipment rinseates, trip blanks, or source blanks on CLP Chain-of-Custody Records as directed by the EPA Region.

10. Enter date of sample collection. ✓

11. Enter time of sample collection in military time. ✓

12. Enter an X in appropriate box for sample designation - composite or grab. ?

13. Samplers must enter their initials next to the samples they collected.

14. List parameters for analysis and the number of containers submitted for each analysis.

15. Enter MS/MSD or MS if sample is for laboratory QC, or other remarks, e.g., sample depth.

16. Record the type of preservative added by reference number and sample pH. Use the remarks column if no space is dedicated to preservative.

17. <u>All</u> samplers must sign in the space provided.

18. The originator checks information entered in 1 through 17 and then signs the top left "Relinquished by" box and enters the current date and time (military).

- Upon completion of the custody record form, the white and pink copies shall be sent with the samples to the laboratory; the yellow copy is retained for the project files. Additional copies will be retained for the project file or distributed as required to the appropriate sample coordinators for CLP work.

19. The laboratory sample custodian receiving the samples checks the sample label information against the custody record form. He or she also checks sample condition and notes anything unusual under "Remarks" on the custody record form. The laboratory custodian receiving custody signs in the adjacent "Received by" box and keeps the pink copy. The white copy is returned to CDM Federal.

5.2 Sample Tags for CLP Projects

Sample tags will be utilized for all samples collected or accepted for CLP projects.

1. All sample tags will be securely attached to the sample bottle. On 80 oz. amber bottles, the tag string may be looped through the ring style handle and tied. On all other containers, it is recommended that the string be looped around the neck of the bottle, then twisted and relooped around the neck until the slack in the string is removed.

2. One tag will be completed for each sample container collected. Each tag will be completed as follows (see Figure 2: EPA CLP Sample Tag) (check with your EPA Regional guidelines for variations):

   - Under the "Remarks" heading, enter the CLP Case Number and record the CLP sample number.
   - Record the Project Code assigned by EPA.
   - Enter the Station Number if applicable.
   - Record the date to indicate the month, day, and year of sample collection.
   - Enter the time (military) of sample collection.
   - Place a check to indicate composite or grab sample.
   - Record the sample location.
   - Samplers must sign in the space provided.
   - Place a check next to "yes" or "no" to indicate if a preservative was added.
   - Under "analyses", place a check next to the parameters for which the sample is to be analyzed. If the desired analysis is not listed, write it in the empty slot. Note: Do not write in the box for "laboratory sample number".

5.3 Sample Shipping

CDM Federal's Standard Operating Procedure 2-5: Packaging and Shipping of Environmental Samples establishes a uniform method for packaging and shipping low level environmental samples.

6.0 RESTRICTIONS/LIMITATIONS

For EPA CLP sampling events, combined chain-of-custody/traffic report forms will be utilized. Refer to Regional guidelines for completing these forms.

7.0 REFERENCES

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.

U.S. Environmental Protection Agency, *Samplers Guide to the Contract Laboratory Program*, EPA/540/P-90/006, December 1990.

Figure 1
EXAMPLE CDM Federal Chain-of-Custody Record

CDM FEDERAL PROGRAMS CORPORATION
A subsidiary of Camp Dresser & McKee Inc.

98 North Washington Street, Suite 200
Boston, MA 02114
617-742-2659

CHAIN OF CUSTODY RECORD

PROJECT NO. #1	FIELD TEAM LEADER #2	LABORATORY AND ADDRESS #3	RECORD ___ OF #4
PROJECT NAME/LOCATION #5			AIRBILL NO. #6

SAMPLE TYPES #7
1. SURFACE WATER
2. GROUNDWATER
3. LEACHATE
4. RINSATE
5. SOIL/SEDIMENT
6. OIL
7. WASTE
8. OTHER ______

PARAMETERS DESIRED — List no. of containers submitted — ANALYSES

PRESERVATIVES ADDED: 1. HCl 2. HNO3 3. H2SO4 4. NaOH 5. Ice only 6. Not Preserved 7. Other ____

SAMPLE NO.	SAMPLE TYPE	19__ DATE	TIME SAMPLED	COMP	GRAB	SAMPLERS INITIALS*	ANALYSES						REMARKS (Note if MS/MSD)	PRESERVATIVES ADDED
#8	#9	#10	#11	#12		#13	#14						#15	#16

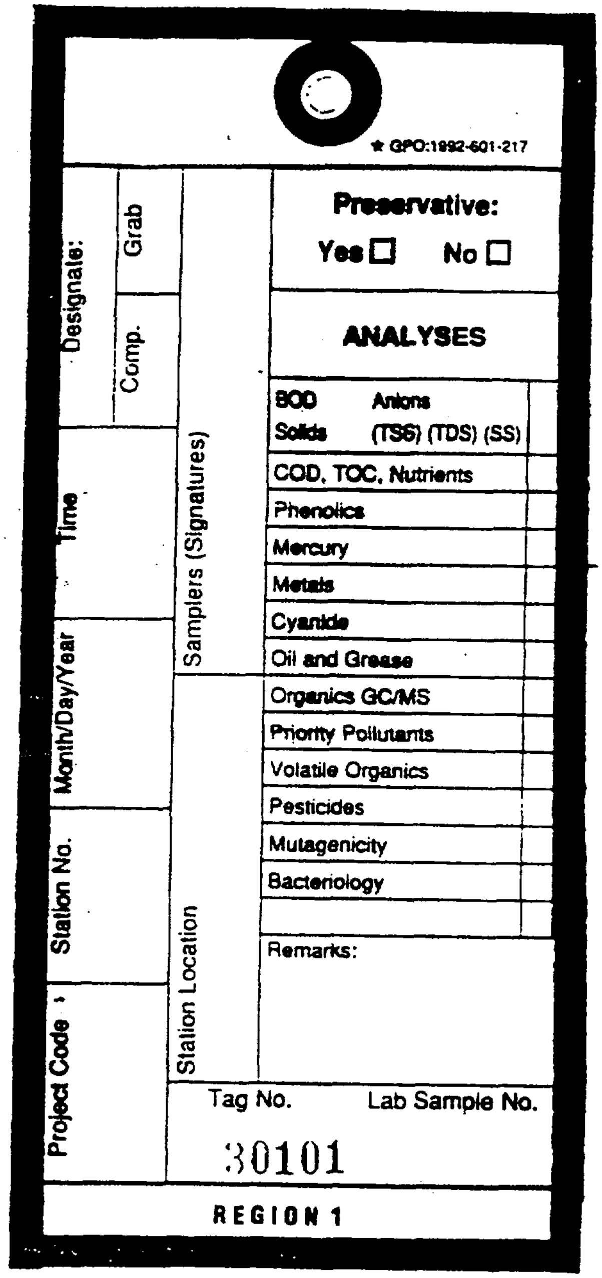
* SAMPLER SIGNATURES: #17

RELINQUISHED BY: (print) (sign) #18	DATE/TIME	RECEIVED BY: (print) (sign) #19	RELINQUISHED BY: (print) (sign)	DATE/TIME	RECEIVED BY: (print) (sign)
RELINQUISHED BY (print) (sign)	DATE/TIME	RECEIVED BY: (print) (sign)	RELINQUISHED BY: (print) (sign)	DATE/TIME	RECEIVED BY: (print) (sign)

DISTRIBUTION: White and yellow copies accompany sample shipment to laboratory; yellow copy retained by laboratory. Pink copy retained by samplers. (10/92)

NOTE: If requested by the client, different Chain-of-Custody records may be used.
Copies of the template for this record may be obtained from the Fairfax Graphics Department.

Figure 2
EXAMPLE EPA CLP Sample Tag


★ GPO:1992-601-217

Designate:
Grab
Comp.
Time
Month/Day/Year
Station No.
Project Code
Samplers (Signatures)
Station Location

Preservative:
Yes ☐ No ☐

ANALYSES

BOD Anions
Solids (TSS) (TDS) (SS)
COD, TOC, Nutrients
Phenolics
Mercury
Metals
Cyanide
Oil and Grease
Organics GC/MS
Priority Pollutants
Volatile Organics
Pesticides
Mutagenicity
Bacteriology

Remarks:

Tag No. Lab Sample No.
30101

REGION 1

NOTE: If requested by the client, different sample tags may be used.

SURFACE SOIL SAMPLING

Prepared: Del R. Baird

Technical Review: Ken Black

QA Review: David O. Johnson

Approved: [signature] 2/24/99
Signature/Date

Issued: [signature] 2/24/99
Signature/Date

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to define the techniques and the requirements for collecting surface soil samples.

2.0 BACKGROUND

2.1 Definitions

Surface Soil - The soil that exists down from the surface approximately one foot (30 centimeters). Depending on application, the soil interval to be sampled will vary.

Grab Sample - A discrete portion or aliquot taken from a specific location at a given point in time.

Composite - Two or more subsamples taken from a specific media and site at a specific point in time. The subsamples are collected and mixed, then a single average sample is taken from the mixture.

Spoon/Scoop - A small stainless steel or Teflon utensil approximately 6 inches in length with a stem-like handle.

Trowel - A small stainless steel or Teflon shovel approximately 6 to 8 inches in length with a slight (approximately 140°) curve across. The trowel has a stem-like handle (for hand operation). Samples are collected with a spooning action.

2.2 Discussion

Surface soil samples are collected to determine the type(s) and level(s) of contamination and are often important to risk assessment. These samples may be collected as part of an investigative plan, site-specific sampling plan, and/or as a screen for "hot spots," which may require more extensive sampling. Sediment(s) and sludge(s) that have been exposed by evaporation, stream rerouting, or any other means are collected by the same methods as those for surface soil(s). Typically, the top 1 to 2 centimeters (cm) of material, including vegetation, are carefully removed before collection of the sample.

Surface soil and exposed sediment or sludge are collected using stainless steel and/or Teflon-lined trowels or scoops.

2.3 Associated Procedures

- CDM Federal SOP 1-2, Sample Custody
- CDM Federal SOP 2-5, Packaging and Shipping of Environmental Samples
- CDM Federal SOP 4-1, Field Logbook Content and Control
- CDM Federal SOP 4-5, Field Equipment Decontamination at Nonradioactive Sites

3.0 RESPONSIBILITIES

Site Manager - The Site Manager is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any other SOPs pertaining to specific media sampling.

Field Team Leader - The Field Team Leader is responsible for ensuring that field personnel collect surface soil samples in accordance with this and other relevant procedures.

4.0 REQUIRED EQUIPMENT

- Insulated cooler and waterproof sealing tape
- Ice bags or "blue ice"
- Latex or appropriate gloves
- Plastic zip-top bags
- Personal protective clothing and equipment
- Stainless steel and/or Teflon-lined spatulas and pans, trays, or bowls
- Stainless steel and/or Teflon-lined trowels or spoons (or equipment as specified in the site-specific plans)

- Plastic sheeting

Additional equipment are discussed in Section 5.2.2, VOC Field Sampling/Preservation Methods.

5.0 PROCEDURES

5.1 Preparation

The following steps must be followed when preparing for sample collection:

1. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan.

2. The collection points shall be stated, located on a site map, and referenced in the field logbook.

3. Processes for verifying depth of sampling must be specified in the site-specific plans.

4. Place clean plastic sheeting on a flat, level surface near the sampling area, if possible, and place equipment to be used on the plastic; place the insulated cooler(s) on separate plastic sheeting. Cover all equipment and supplies with clean plastic sheeting when not in use.

5. A clean, decontaminated trowel, scoop, or spoon will be used for each sample collected. Other equipment may be used (e.g., shovels) if constructed of stainless steel.

5.2 Collection

The following general steps must be followed when collecting surface soil samples:

1. Surface soil samples are normally collected from the least-contaminated to the most-contaminated areas. Stay outside a specific sampling location until all samples are collected at that location.

2. Document the sampling events, recording the information in the designated field logbook. Document any and all deviations from SOPs in the field logbook and include rationale for changes. See CDM Federal SOP 4-1.

3. Carefully remove stones, vegetation, snow, etc. from the sampling location surface.

4. First collect sample portions or aliquots for volatile analyses as well as any other samples that would be degraded by aeration. Follow with collection of samples for other analyses.

6. Decontaminate sampling equipment between locations. See CDM Federal SOP 4-5.

5.2.1 Method for Collecting Samples for Volatile Organic Compound (VOC) Analysis

The requirements for collecting grab samples of surface soil for VOCs or other samples degraded by aeration are as follows:

1. VOC samples shall be collected with the least disturbance possible.

2. VOC samples shall be collected as grab samples; however, the method of collection will vary from site to site, based on data quality objectives and the degree of known or suspected contamination.

3. Label the sample containers with the appropriate information. Secure the label, covering it with a piece of clear tape.

4. Use a clean stainless steel or Teflon-lined trowel or spoon (or tube) to collect sufficient material in one grab, over the required sampling interval, to fill the sample containers.

5. With the aid of a clean stainless steel spatula, quickly fill the sample containers directly from the sampling device, removing stones, twigs, grass, etc., from the sample. Fill the containers as full and compact as possible to minimize headspace.

6. Immediately secure the Teflon-lined caps on the sample container.

7. Wipe the containers clean with a clean Kimwipe or paper towel.

8. Place the containers in individual zip-top plastic bag(s) and seal the bag(s).

9. Pack all samples as required. Include properly completed documentation, and affix signed and dated custody seals to the cooler lid.

5.2.2 Field Sampling/Preservation Methods

The following four sections contain SW 846 methods for sampling and field preservation. These methods include EN CORE™ Sampler Method for low level detection limits, EN CORE™ Sampler Method for high level/detection limits/screening, Acid preservation, and Methanol preservation. These methods may be used if required by the EPA Region, client, or governing sample plan. These methods are very detailed and contain equipment requirements at the beginning of each section.

Note: Some variations from these methods may be required depending on the contracted analytical laboratory, such as sample volume.

5.2.2.1 EN CORE™SAMPLER COLLECTION FOR LOW LEVEL ANALYSES (≥1 UG/KG)

<u>EN CORE™Sampling Equipment Requirements</u>

The following equipment is required for low level analysis:

- Two 5g samplers.
- One 25g sampler or one 5g sampler for screening and or high level analysis. (The sampler
- size used will be dependent on who is doing the sampling and who is doing the laboratory
- analysis).
- One dry weight cup.
- One T-handle.
- Paper toweling.

<u>EN CORE™ Sampling Steps for Low Level Analysis</u>

1. Remove sampler and cap from package and attach T-handle to sampler body.

2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.

3. Use paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.

4. Push cap on with a twisting motion to attach cap.

5. Fill out label and attach to sampler.

6. Repeat procedure for the other two samplers.

7. Collect dry weight sample.

8. Store samplers at 4 degrees Celsius.

Ship sample containers with plenty of ice to the laboratory within 40 hours of collection.

5.2.2.2 ACID PRESERVATION SAMPLING FOR LOW LEVEL ANALYSES (≥1 UG/KG)

Acid Preservation Sampling Equipment Requirements

The following equipment and supplies are required for field Acid preservation:

- One 40mL VOA vial with acid preservation (for field testing of soil pH). Two pre-weighed 40mL VOA vials with acid preservative and stir bar (for lab analysis)
- Two pre-weighed 40mL VOA vials with water and stir bar (in case samples effervesces).
- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a
- pre-weighed vial that contains methanol (for screening sample and/or high level analysis).
- One dry weight cup.
- One 2oz jar with acid preservative (in case additional acid is needed due to high
- soil pH).
- One scoop capable to deliver about one gram of solid sodium bisulfate.
- pH paper.
- Weighing balance that weighs to 0.01 g (with an accuracy of ±0.1g).
- Set of balance weights used in daily balance calibration.
- Gloves for working with pre-weighed sample vials.
- Paper toweling.
- Sodium bisulfate acid ($NaHSO_4$) acid.
- A cutoff plastic syringe or other coring device to deliver 5 g or 25g of soil.

Testing Effervescing Capacity of Soils

Soils must be tested with acid to determine the amount of effervescing that will occur when preserved with acid. Effervescing will drive off VOCs as well as create a very high pressure in a sealed vial which could explode. The following steps will provide information on the effervescing capacity of the soil.

1. Place~5g of soil into a vial that contains acid preservative and no stir bar.

2. Do not cap this vial as it may EXPLODE upon interaction with the soil.

3. Observe the sample for gas evolution (due to carbonates in the soil).

4. If vigorous or sustained gas evolution occurs; then acid preservation is not acceptable to preserve the sample.

- In this case the samples need to be collected in the VOA vials with only water and a stir bar. The vials with acid preservative CANNOT be used.

5. If a small amount or no gas evolution occurs: then acid preservation is acceptable to preserve the sample. Keep this testing vial for use in the buffering testing detailed below.

- In this case the samples need to be collected in the VOA vials with the acid preservative and a stir bar.

Testing Buffering Capacity of Soils

The soils must be tested to determine the quantity of acid that is required to reach a less than 2 pH reading. The following steps will assist in determining this quantity.

1. If acid preservation is acceptable for sampling soils then the sample vial that was used in the effervescing testing can be used here for the buffering testing.

2. Cap the vial that contains 5g of soil, acid preservative and no stir bar from step 1 in the effervescing testing.

3. Shake the vial gently to attempt to make a homogenous solution.

4. When done, open the vial and check the pH of the acid solution with the pH paper.

- If the pH paper reads below 2 then the sampling can be done in the two pre-weighed 40mL VOA trials with the acid preservative and stir bar. Since the pH was below 2, it is not necessary to add additional acid to the vials.

- If the pH paper reads above 2, then additional acid needs to be added to the sample vial.

5. Use the jar with the solid sodium bisulfate acid and add another one gram of acid to the sample.

6. Cap the vial and shake thoroughly again.

7. When done, open the vial and check the pH of the acid solution with a new piece of paper.

- If the pH paper reads below 2 then the sampling can be done in the two pre-weighed 40mL VOA trials with the acid preservative and stir bar and one extra gram of acid.

- Make a note of the extra gram of acid needed so the same amount of extra acid can be added to the vials the lab will analyze.

- If the pH paper reads above 2, then add another gram of acid and repeat this procedure one more time.

Now that the soil chemistry has been determined the actual sampling can occur. The procedure stated below assumes the correct vials are used based on the guidance discussed.

Sample Preservation Steps

1. Wear gloves during all handling of pre-weighed vials.

2. Quickly collect a 5g sample using a cut off plastic syringe or other coring device designed to deliver 5g of soil from a freshly exposed surface of soil.

3. Carefully wipe exterior of sample collection device with clean paper toweling.

4. Quickly transfer to the appropriate VOA trial, extruding with caution so that the solution does not splash out of the vial.

5. Add more acid if necessary (this is based on the buffering testing discussed in the previous section).

6. Use the paper toweling and quickly remove any soil off the vial threads.

7. Cap vial and weigh the jar to the nearest 0.01 g.

8. Record exact weight on sample label.

9. Repeat sampling procedure for the duplicate VOA vial.

10. Weigh the vial with methanol preservative in it to the nearest 0.01g. If the weight of the vial with methanol varies by more than 0.01g from the original weight recorded on the vial-discard the vial. If the weight is within tolerance it can be used for soil preservation below.

11. Take the empty jar or the jar that contains the methanol preservative.

12. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size is dependent on who is doing the sampling and who is doing the laboratory analysis.

13. Carefully wipe the exterior of the collection device with clean paper toweling.

14. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol be careful not to splash the methanol outside of the vial. Again, the type of jar received is dependent on who is doing the laboratory analysis.

15. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided-using only one vial of methanol preservative per sample jar.

16. Use the paper toweling and remove any soil off of the vial threads and cap the jar.

17. Weigh the jar with the soil in it to the nearest 0.01g and record the weight on the sample label.

18. Collect dry weight sample-fill container.

19. Store samples at 4 degrees Celsius.

20. Ship sample containers with plenty of ice and per Department of Transportation (DOT) regulations (CORROSIVE. FLAMMABLE LIQUID. POISON) to the laboratory.

5.2.2.3 EN CORE SAMPLER COLLECTION FOR HIGH LEVEL ANALYSES (≥200 UG/KG)

EN CORE™ Sampling Equipment Requirements

The following equipment is required for high level analysis:

- One 25g sampler or one 5g sampler (The sampler size used will be dependent on who is doing the sampling and who is doing the laboratory analysis).
- One dry weight cup.
- One T-handle.
- Paper toweling.

EN CORE™Sampling Steps for High Level Analysis

1. Remove sample and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.
3. Use paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.
4. Push cap on with a twisting motion to attach cap.
5. Fill out label and attached to sampler.
6. Collect dry weight sample.
7. Store samplers at 4 degrees Celsius.
8. Ship sample containers with plenty of ice to the laboratory within 40 hours of collection.

5.2.2.4 METHANOL PRESERVATION SAMPLING FOR HIGH LEVEL ANALYSES (≥200 UG/KG)

Methanol Preservation Sampling Equipment Requirements

- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol (laboratory grade).
- One dry weight cup.
- Weighing balance that accurately weighs to 0.01g (with accuracy of ±0.1g).
- Set of balance weights used in daily balance calibration.
- Latex gloves.
- Paper towel.

- Cutoff plastic syringe or other coring device to deliver 5g or 25g of soil.

<u>Sampling Preservation Steps</u>

1. Wear gloves during all handling of pre-weighed vials.

2. Weigh the vial with methanol preservative in it to the nearest 0.01g. If the weight of the vial with methanol varies by more than 0.01g from the original weight recorded on the vial-discard the vial. If the weight is within tolerance it can be used for soil preservation/collection below.

3. Take the empty jar or the jar that contains the methanol preservative.

4. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size used is dependent on who is doing the sampling and who is doing the laboratory analysis.

5. Carefully wipe the exterior of the collection device with clean paper toweling.

6. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol be careful not to splash the methanol outside of the vial. Again, the type of jar used is dependent on who is doing the laboratory analysis.

7. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided-using only one vial of methanol preservative per sample jar.

8. Using the paper toweling-remove any soil off of the vial threads and cap the jar.

9. Weigh the jar with the soil in it to the nearest 0.01g and record the weight on the sample label.

10. Collect dry weight sample-fill container.

11. Store samples at 4 degrees Celsius.

12. Ship sample containers with plenty of ice and per DOT regulations (CORROSIVE. FLAMMABLE LIQUID. POISON) to the laboratory.

5.2.3 Method for Collecting Samples for Nonvolatile Organic or Inorganic Compound Analysis

The requirements for collecting samples of surface soil for nonvolatile organic or inorganic analyses are as follows:

1. Label each sample container with the appropriate information. Secure the label by covering it with a piece of clear tape.

2. Use a decontaminated stainless steel or Teflon-lined trowel or spoon to obtain sufficient sample from the required interval and subsampling points, if necessary, to fill the specified sample containers.

3. Empty the contents of each fill of the sampling device directly into a clean stainless steel or Teflon-lined tray or bowl.

4. Homogenize the sample by mixing with a spoon, spatula, or trowel.

5. Use the spoon, spatula, or trowel to distribute the uniform mixture into the labeled sample containers. Fill organic sample containers first, then inorganics.

6. Secure the appropriate cap on each container immediately after filling it.

7. Wipe the sample containers clean with a clean Kimwipe or paper towel.

8. Place sample containers in individual zip-top plastic bags and seal the bags.

9. Pack all samples as required. Include properly completed documentation, and affix custody seals to the cooler lid.

10. Decontaminate sampling equipment according to CDM Federal SOP 4-5.

6.0 RESTRICTIONS/LIMITATIONS

When grab sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration, it is necessary to minimize sample disturbance and, hence, analyze loss. The representativeness of this sample, however, is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

7.0 REFERENCES

U.S. Department of Energy, Hazardous Waste Remedial Actions Program, *Quality Control Requirements For Field Methods,* DOE/HWP-69/R1, July 1990 or current revision.

U.S. Department of Energy, Hazardous Waste Remedial Actions Program, *Standard Operating Procedures For Site Characterizations*, DOE/HWP-100, July 1990 or current revision.

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods,* EPA/540/P-87/001, December 1987 or current revision.

U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846),* Third Edition, November 1986, (as amended by Update III, June 1997). Method 5035: Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples.

PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES

Prepared: 10/5/95 Signature/Date
Technical Review: 10/19/95 Signature/Date
QA Review: Teresa A. Neli 10/20/95 Signature/Date
Approved: 11/8/95 Signature/Date
Issued: Rose Mary Ellersick 11/14/95 Signature/Date

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to establish packaging and shipping requirements and guidelines for environmental sample shipping.

2.0 BACKGROUND

2.1 Definitions

Environmental Sample - An Environmental Sample is any sample that has less than reportable quantities for any hazardous constituents according to Department of Transportation (DOT) regulations promulgated in 49 CFR - Part 172.

Chain of Custody Seal - Narrow strip of adhesive paper used to demonstrate that no tampering has occurred.

2.2 Discussion

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis.

2.3 Associated Procedure

- CDM Federal SOP 1-2, Sample Custody

3.0 RESPONSIBILITIES

Field Team Leader - The Field Team Leader is responsible for ensuring that packaging and sampling procedures are conducted in accordance with this SOP. The Field Team Leader is also responsible for ensuring that laboratory analysis of samples is properly coordinated by CDM Federal.

4.0 REQUIRED EQUIPMENT

- Coolers with return address of CDM Federal office
- Heavy-duty plastic garbage bags
- Plastic zip-top bags, small and large
- Clear Tape
- Fiber tape
- Duct tape
- Vermiculite (or equivalent)*
- Bubble wrap (optional)
- Ice
- Chain-of-Custody seals
- Completed Chain-of-Custody record or CLP custody records if applicable
- Completed Bill of Lading
- "This End Up" and directional arrow labels

* Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

5.0 PROCEDURES

The following steps must be followed when packing sample bottles and jars for shipment:

1. Verify the samples undergoing shipment meet the definition of "Environmental Sample" and are not a hazardous material as defined by DOT. Professional judgement and/or consultation with the appropriate Health and Safety Coordinator or the Health and Safety Manager should be observed.

2. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape. Line the cooler with a large heavy-duty plastic garbage bag.

3. Be sure the caps on all bottles are tight (will not leak); check to see that labels and chain-of-custody records are completed properly (SOP 1-2, Sample Custody).

4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three VOA vials may be packed in one bag. Bottles may be wrapped in bubble wrap. Optionally, place three to six VOA vials in a quart metal can and then fill the can with vermiculite or equivalent. **Note:** Trip blanks must be included in coolers containing VOA samples.

5. Place two to four inches of vermiculite (or equivalent) into a cooler that has been lined with a trash bag, and then place the bottles and cans in the bag with sufficient space to allow for the addition of more packing material between the bottles and cans. It is preferable to place glass sample bottles and jars into the cooler vertically. Due to the strength properties of a glass container, there is much less chance for breakage when the container is packed vertically rather than horizontally.

6. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place the ice bags on top of and/or between the samples. Several bags of ice are required (dependant on outdoor temperature, staging time, etc.) to maintain the cooler temperature at less than or equal to 4° Centigrade. Fill all remaining space between the bottles or cans with packing material. Securely fasten the top of the large garbage bag with fiber or duct tape.

7. Place the completed Chain-of-Custody Record or the CLP Traffic Report Form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler's lid, and then close the cooler.

8. Fiber tape shall be wrapped around each end of the cooler two times, and completed Chain-of-Custody seals affixed to the top opposite sides of the cooler, half on the fiber tape so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with fiber tape; place clear tape over custody seals.

9. The shipping container lid must be marked "THIS END UP" and arrow labels which indicate the proper upward position of the container should be affixed to the cooler. A label containing the name and address of the shipper (CDM Federal) shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted to be on the outside of the container used to transport environmental samples and shall not be used. The name and address of the laboratory shall be placed on the container, or when shipping by common courier, the Bill of Lading shall be completed and attached to the lid of the shipping container.

6.0 RESTRICTIONS/LIMITATIONS

The holding times for the samples packed for shipment must not be exceeded. It is recommended that samples be packed in time to be shipped nightly for overnight delivery. Use caution when shipping samples for weekend delivery; make arrangements with laboratory before sending samples.

7.0 REFERENCES

U.S. Environmental Protection Agency, *Sampler's Guide to the Contract Laboratory Program*, EPA/540/P-90/006, December 1990.

U.S. Environmental Protection Agency, Region IV, *Standard Operating Procedures and Quality Assurance Manual*, February 1991.

Prepared: Del Baird

Technical Review: Jackie Mosher

QA Review: David O. Johnson

Approved: 2/23/99
Signature/Date

Issued: 2/24/99
Signature/Date

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to set CDM Federal criteria for content entry and form of field logbooks.

2.0 BACKGROUND

2.1 Definitions

Biota - The flora and fauna of a region.

Decontamination - To remove contaminants from field sampling equipment that might bias analytical results.

Magnetic Declination Corrections - Compass adjustments to correct for the angle between magnetic north and geographical meridians.

2.2 Discussion

Information recorded in field logbooks include observations, data, calculations, time, weather, description of the data collection activity, methods, instruments, and results. Additionally, the logbook may contain descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

3.0 RESPONSIBILITIES

Field Team Leader (FTL) - The FTL is responsible for ensuring the nature and form of data entries are conducted in accordance with this procedure.

Site Personnel - All CDM Federal employees who make entries in field logbooks during on-site activities are required to read this procedure prior to engaging in this activity. The FTL will assign field logbooks to site personnel who will be responsible for their care and maintenance.

4.0 REQUIRED EQUIPMENT

- Site-specific plans
- Field notebook
- Indelible black or blue ink pen
- Ruler or similar scale (in some circumstances)

5.0 PROCEDURES

5.1 Preparation

In addition to this SOP, site personnel responsible for maintaining logbooks must be familiar with other pertinent CDM Federal and site SOPs. These should be consulted as necessary to obtain specific information about equipment and supplies, health and safety, sample collection, packaging, decontamination, and documentation. These procedures should be located at the field office.

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered prior to initial use of the logbook. Prior to use in the field, each logbook will be marked with a specific document control number issued by the document control administrator, if required by the QIP. Not all contracts require document control numbers. The following information shall be recorded on the cover of the logbook:

- Field Logbook Document Control Number
- Activity (if the logbook is to be activity-specific)
- Name of CDM Federal contact and phone number(s)
- Start date

The first few (approximately five) pages of the logbook will be reserved for a table of contents. Mark the first page with the heading and enter the following:

TABLE OF CONTENTS

Date/Description	Page
(Start Date)/Reserved for TOC	1-5

The remaining pages of the Table of Contents will be designated as such with "TOC" written on the top center of each page.

5.2 Operation

The following is a list of requirements that must be followed when using a logbook:

- Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are specified by an activity-specific plan, this information need not be duplicated in the logbook. However, any forms used to record site information must be referenced in the logbook.

- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.

- Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion. Take care to not obliterate what was written previously.

- Do not remove any pages from the book.

- Record as much information as possible.

Specific requirements for field logbook entries include:

- Initial and date each page
- Sign and date the final page of entries for each day
- Initial and date all changes
- Multiple authors must sign out the logbook by inserting the following:

  Above notes authored by:
  - (Sign name)
  - (Print name)
  - (Date)

- A new author must sign and print his/her name before additional entries are made
- Draw a diagonal line through the remainder of the final page at the end of the day
- Record the following information on a daily basis:

  - Date and time
  - Name of individual making entry

- Names of field team and other persons on-site
- Description of activity being conducted including station (i.e., well, boring, sampling location number) if appropriate
- Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction, and speed) and other pertinent data
- Level of personal protection to be used
- Serial numbers of instruments
- Required calibration information
- Serial/tracking numbers on documentation (e.g., carrier airbills)

Entries into the field logbook shall be preceded with the time (written in military units) of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In these cases, the logbook must reference the automatic data record or form.

At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. Use a compass (include a reference to magnetic declination corrections), scale, or nearby survey markers, as appropriate. A sketch of station location may be warranted. All maps or sketches made in the logbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketches be oriented so that north is toward the top of the page.

Other events and observations that should be recorded include:

- Changes in weather that impact field activities
- Deviations from procedures outlined in any governing documents. Also record the reason for any noted deviation.
- Problems, downtime, or delays
- Upgrade or downgrade of personal protection equipment

5.3 Post-Operation

To guard against loss of data due to damage or disappearance of logbooks, completed pages shall be periodically photocopied (weekly, at a minimum) and forwarded to the field or project office. Other field records shall be photocopied and submitted regularly and as promptly as possible to the office. When possible, electronic media such as disks and tapes should be copied and forwarded to the office.

At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated, and that corrections were made properly (single lines drawn through incorrect information, then initialed and dated). The completed logbook shall be submitted to the records file.

6.0 RESTRICTIONS/LIMITATIONS

Field logbooks constitute the official record of on-site technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by CDM Federal personnel and their subcontractors. They are documents that may be used in court to indicate and defend dates, personnel, procedures, and techniques employed during site activities. Entries made in these notebooks should be factual, clear, precise, and as non-subjective as possible. Field logbooks, and entries within, are not to be utilized for personal use.

7.0 REFERENCES

Sandia National Laboratories, *Procedure for Preparing, Sampling and Analysis Plan, Site-Specific Sampling Plan, and Field Operating Procedures*, QA-02-03, Albuquerque Environmental Program Department 3220, Albuquerque, New Mexico, 1991.

Sandia National Laboratories, Division 7723, *Field Operation Procedure for Field Logbook Content and Control*, Environmental Restoration Department, Albuquerque, New Mexico, 1992.

FIELD EQUIPMENT DECONTAMINATION AT NONRADIOACTIVE SITES

Prepared: Susan Flakus   Technical Review: Dave Schroeder

QA Review: David O. Johnson   Approved: 3/13/98
Signature/Date

Issued: 3/13/98
Signature/Date

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to describe the procedures required for decontamination of field equipment.

2.0 BACKGROUND

2.1 Definitions

Clean - Free of visible contamination and when decontamination has been completed in accordance with this SOP.

Cross-Contamination - The transfer of contaminants through equipment or personnel from the contamination source to less contaminated or noncontaminated samples or areas.

Decontamination - The process of rinsing or otherwise cleaning the surfaces of equipment to rid them of contaminants and to minimize the potential for cross contamination of samples or exposure of personnel.

2.2 Discussion

Decontamination of field equipment is necessary to ensure the quality of samples by preventing cross contamination. Further, decontamination reduces health hazards and prevents the spread of contaminants off-site.

3.0 RESPONSIBILITIES

Field Team Leader - The Field Team Leader (FTL) ensures that field personnel are trained in the performance of this procedure and that decontamination is conducted in accordance with this procedure. The FTL may also be required to collect and document rinsate samples to provide quantitative verification that these procedures have been correctly implemented.

4.0 REQUIRED EQUIPMENT

- Stiff-bristle scrub brushes
- Plastic buckets and troughs
- Laboratory-grade detergent (low phosphate)
- Nalgene or Teflon Sprayers or wash bottles or 2- to 5-gallon, manual-pump sprayer (pump sprayer material must be compatible with the solution used)
- Plastic sheeting
- Disposable wipes or rags
- Potable water and/or deionized water and/or American Society for Testing and Materials (ASTM) Type II or better, as defined by ASTM Standard Specification for Reagent Water, Standard D 1193-77 (re-approved 1983)*
- Gloves, safety glasses, and other protective clothing as specified in the site-specific health and safety plan
- High-pressure pump with soap dispenser or steam-spray unit (for large equipment only)
- Appropriate decontamination solutions pesticide grade or better and traceable to a source (e.g. 10% and/or 1% nitric acid (HNO_3), acetone, methanol, isopropanol, hexane)
- Tools for equipment assembly and disassembly (as required)
- 55-gallon drums or tanks (as required)
- Pallets for drums or tanks holding decontamination water (as required)

* Potable, deionized, and ASTM Type II water may be required to be tested for contaminants before use. Check field plan for requirements.

5.0 PROCEDURES

All reusable equipment (non-dedicated) used to collect, handle, or measure samples will be decontaminated before coming into contact with any sample. Decontamination of equipment will occur either at the central decontamination station or at portable decontamination stations set up at the sampling location, drill sites, or monitoring well locations. The centrally located decontamination station will include an appropriately sized bermed area on which equipment decontamination will occur and shall be equipped with a collection system and storage vessels. In certain circumstances, berming is not required when small quantities of water are being generated and for some short duration field activities (i.e., pre-remedial sampling).

The decontamination area will be constructed so that contaminated water is either collected directly into appropriate containers (5-gallon buckets or steel wash tubs) or within the berms of the decontamination area which then drains into a collection system. Water from the collection system will be pumped into 55-gallon drums or portable tanks for storage. Typically, decontamination water will be staged until sampling results or waste characterization results are obtained and evaluated and the proper disposition of the waste is determined. The exact procedure for decontamination waste disposal should be discussed in the field plan. Also, decontamination fluids, such as solvents may need to be segregated from other investigation derived wastes.

All items that will come into contact with potentially contaminated media will be decontaminated before use and between sampling and/or drilling locations. If decontaminated items are not immediately used, they will be covered either with plastic or aluminum foil depending on the size of the item. All decontamination procedures for the equipment being used are as follows:

General Guidelines

- Potable and deionized water should be free of all contaminants of concern. Following the field plan, analytical data from the water source may be required. If required, either existing analytical data from the water source supplier (i.e., municipality, bottled water company, deionized water producer) may be obtained or chemical testing may be performed on the selected source.

- Soap used in the soap and water rinse step will be a low phosphate detergent.

- Sampling equipment that has come into contact with oil and grease will be cleaned with methanol or other approved alternative to remove the oily material. This may be followed by a hexane rinse and then another methanol rinse. Regulatory or client requirements will be stated in the field plan.

- Decontaminated equipment will be allowed to air dry before being used.

- Documentation for all cleaning will be recorded in the appropriate logbook.

- All solvents will be pesticide grade or better and traceable to a source. The corresponding lot numbers will be recorded in the appropriate logbook.

- Gloves, boots, safety glasses, and any other personnel protective clothing and equipment will be used as specified in the site-specific health and safety plan.

5.1 Heavy Equipment Decontamination

Heavy equipment includes drilling rigs and backhoes. Follow these steps when decontaminating this equipment:

1. Establish a decontamination area with berms that is large enough to fully contain the equipment to be cleaned. If available, an existing wash pad or appropriate paved and bermed area may be utilized; otherwise, use one or more layers of heavy plastic sheeting to cover the

ground surface and berms. All decontamination pads should be upwind of the area to investigated.

2. With the rig in place, spray areas (rear of rig or backhoe) exposed to contaminated soils using a hot water high-pressure sprayer. Be sure to spray down all surfaces, including the undercarriage.

3. Use brushes, and low phosphate detergent and potable water to remove dirt whenever necessary.

4. Remove equipment from the decontamination pad and allow it to air dry before returning it to the work site.

5. Record equipment type, date, time, and method of decontamination in the appropriate logbook.

6. After decontamination activities are completed, collect all contaminated waste water, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal as detailed in the field plan. Liquids and solids must be drummed separately.

5.2 Downhole Equipment Decontamination

Downhole equipment decontamination includes hollow-stem augers, drill pipes, casings, screens, etc. Follow these steps when decontaminating this equipment:

1. Set up a centralized decontamination area, if possible. This area should be set up to collect contaminated rinse waters and to minimize the spread of airborne spray.

2. Set up a "clean" area upwind of the decontamination area to receive cleaned equipment for air drying. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces on which decontaminated equipment is to be placed. All decontamination pads should be upwind of any areas under investigation.

3. Place the object to be cleaned on aluminum foil or plastic-covered wooden sawhorses or other supports.

4. Using low phosphate detergent and potable water in the hot water high-pressure sprayer (or steam unit), spray the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and gaps especially well. Use a brush, if necessary, to dislodge dirt.

5. If using soapy water, rinse the equipment using clean, potable water. If using hot water, the rinse step is not necessary if the hot water does not contain a detergent. If the hot water contains a detergent, this final clean water rinse is required.

6. Using the manual-pump sprayer, rinse the equipment thoroughly with deionized water (ASTM Type II or better).

7. Remove the equipment from the decontamination area and place in a clean area upwind to air dry.

8. Record equipment type, date, time, and method of decontamination in the appropriate logbook.

9. After decontamination activities are completed, collect all contaminated waste waters, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

5.3 Sampling Equipment Decontamination

Sampling equipment includes split spoons, spatulas, and bowls used for sample homogenization that directly contact sample media. Follow these steps when decontaminating this equipment:

1. Set up a decontamination line on plastic sheeting. The decontamination line should progress from "dirty" to "clean" and have an area located upwind for drying decontaminated equipment. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or the surfaces on which decontaminated equipment is to be placed for drying.

2. Before washing, disassemble any items that might trap contaminants internally. Do not reassemble these items until decontamination and air drying are complete. Wash items thoroughly in a bucket of low phosphate detergent and potable water. Use a stiff-bristle brush to dislodge any gross contamination (soil or debris).

3. Rinse the item in potable water. Rinse water should be replaced as needed, generally when cloudy.

4. Using a hand sprayer, wash bottles, or manual-pump sprayer, rinse the item with deionized water (ASTM Type II or better).

5. If required by the site-specific field plans, rinse the item with 10% nitric acid (for stainless steel, glass, plastic, and Teflon), or 1% nitric acid (for items made of low-carbon steel) followed by a deionized water (ASTM Type II or better) rinse.

   NOTE: Care should be taken not to get nitric acid on skin or clothing. This step should not be used unless required by sampling needs as dictated in the field plan.

   CAUTION: Do not allow nitric acid to contact methanol or hexane. Contain nitric acid waste separate from organic solvents.

6. If sampling for organic analytes, rinse the item with methanol or approved organic solvent.

7. Rinse the item with deionized water (ASTM Type II or better).

8. If required by the field plan, when sampling for polar organic compounds such as pesticides, polychlorinated biphenyls (PCBs), and fuels, rinse the item with hexane or approved alternatives, followed by a second methanol rinse.

9. Allow the item to air dry completely.

10. After drying, wrap the clean item in plastic wrap or in aluminum foil, shiny side out.

11. Record equipment type, date, time, and method of decontamination in the appropriate logbook.

12. After decontamination activities are completed, collect all contaminated waters, used solvents and acids, plastic sheeting, and disposable gloves, boots, and clothing. Place contaminated items in properly labeled drums for disposal. Liquids and solids must be drummed separately. (Refer to site-specific plans for labeling and waste management requirements).

5.4 Pump Decontamination

Follow these steps when decontaminating pumps:

1. Set up the decontamination area and separate "clean" storage area using plastic sheeting to cover the ground, tables, and other porous surfaces. Set up three 55-gallon drums and one or more containers of ASTM Type II water (or as specified in the field plan) with one drum containing dilute (nonfoaming) soapy water, the second drum containing potable water, and the third drum receiving waste water.

2. The pump should be set up in the same configuration as for sampling. Submerge the pump intake (or the pump, if submersible) and all downhole-wetted parts (tubing, piping, foot valve) in the soapy water of the first drum. Place the discharge outlet in the waste water drum above the level of the waste water. Pump soapy water through the pump assembly until it discharges to the waste drum.

3. Move the pump assembly to the potable water drum while leaving discharge outlet in the waste drum. All downhole-wetted parts must be immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.

4. Move the pump intake to the ASTM Type II water can. Pump the ASTM Type II water through the pump assembly. Usually, three pump-and-line-assembly volumes will be required.

5. Decontaminate the discharge outlet by hand following the steps outlined in Section 5.3.

6. Remove the decontaminated pump assembly to the "clean" area and allow it to air dry upwind of the decontamination area. Intake and outlet orifices should be covered with aluminum foil to prevent the entry of airborne contaminants and particles.

7. Record the equipment type, serial number, date, time, and method of decontamination in the appropriate logbook.

5.5 Instrument Probe Decontamination

Instrument probes used for field instruments such as pH meters, conductivity meters etc. will be decontaminated between samples and after use with ASTM type II, or better, water.

5.6 Waste Disposal

Refer to site-specific plans for waste disposal requirements. The following are guidelines for disposing of wastes:

1. All wash water, rinse water, and decontamination solutions that have come in contact with contaminated equipment are to be handled, packaged, labeled, marked, stored, and disposed of as investigation-derived waste.

2. Small quantities of decontamination solutions may be allowed to evaporate to dryness.

3. If large quantities of used decontamination solutions will be generated, it may be best to separate each type of waste in a separate container. This may permit the disposal of wash

water and rinse water onsite or in a sanitary sewage treatment plant rather than as a hazardous waste. If an industrial waste water treatment plant is available onsite, the disposal of acid solutions and solvent-water solutions <u>may</u> be permitted.

4. Unless required, plastic sheeting and disposable protective clothing may be treated as a solid, nonhazardous waste.

6.0 RESTRICTIONS/LIMITATIONS

Nitric acid and polar solvent rinses are necessary only when sampling for metals or organics respectively. These steps should not be used unless required because of acid burn and ignitability hazards.

If the field equipment is not allowed to air dry properly before use, volatile organic residue which interferes with the analysis may be detected in the samples. The occurrence of residual organic solvents is often dependent on the time of year sampling is conducted; in the summer, volatilization is rapid and in the winter, volatilization is slow. Check with your EPA region, state and client for approved decontamination solvents.

7.0 REFERENCES

Department of Energy, Hazardous Waste Remedial Actions Program, *Standard Operating Procedures For Site Characterization*, DOE/HWP-100, July 1990.

Department of Energy, Hazardous Waste Remedial Actions Program, *Quality Control Requirements For Field Methods*, DOE/HWP-69/RI.

American Society for Testing and Materials, *Standard Practice for Decontamination of Field Equipment at Nonradioactive Waste Sites*, ASTM D5088-90, June 29, 1990.

U.S. Environmental Protection Agency, Region II, "*CERCLA Quality Assurance Manual*, Revision 1, 1989.

U.S. Environmental Protection Agency, Region IV, *Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual*, 1986.

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001.1, 1987.

APPENDIX C

LABORATORY ANALYTICAL METHODS

ASBESTOS and OTHER FIBERS by PCM 7400

Various MW: Various CAS: Various RTECS: Various

METHOD: 7400, Issue 2	**EVALUATION: FULL**	**Issue 1: Rev. 3 on 15 May 1989** **Issue 2: 15 August 1994**

OSHA : 0.1 asbestos fiber (> 5 µm long)/cc; 1 f/cc/30 min excursion; carcinogen
MSHA: 2 asbestos fibers/cc
NIOSH: 0.1 f/cc (fibers > 5 µm long)/400 L; carcinogen
ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, fibers/cc; carcinogen

PROPERTIES: solid, fibrous, crystalline, anisotropic

SYNONYMS [CAS #]: actinolite **[77536-66-4]** or ferroactinolite **[15669-07-5]**; amosite **[12172-73-5]**; anthophyllite **[77536-67-5]**; chrysotile **[12001-29-5]**; serpentine **[18786-24-8]**; crocidolite **[12001-28-4]**; tremolite **[77536-68-6]**; amphibole asbestos **[1332-21-4]**; refractory ceramic fibers **[142844-00-6]**; fibrous glass.

SAMPLING		**MEASUREMENT**	
SAMPLER:	FILTER (0.45- to 1.2-µm cellulose ester membrane, 25-mm; conductive cowl on cassette)	**TECHNIQUE:**	LIGHT MICROSCOPY, PHASE CONTRAST
		ANALYTE:	fibers (manual count)
FLOW RATE*:	0.5 to 16 L/min	**SAMPLE PREPARATION:**	acetone - collapse/triacetin - immersion
VOL-MIN*: **-MAX*:**	400 L @ 0.1 fiber/cc (step 4, sampling) *Adjust to give 100 to 1300 fiber/mm^2	**COUNTING RULES:**	described in previous version of this method as "A" rules [1,3]
SHIPMENT:	routine (pack to reduce shock)	**EQUIPMENT:**	1. positive phase-contrast microscope 2. Walton-Beckett graticule (100-µm field of view) Type G-22 3. phase-shift test slide (HSE/NPL)
SAMPLE STABILITY:	stable		
BLANKS:	2 to 10 field blanks per set	**CALIBRATION:**	HSE/NPL test slide
ACCURACY		**RANGE:**	100 to 1300 fibers/mm^2 filter area
RANGE STUDIED:	80 to 100 fibers counted	**ESTIMATED LOD:**	7 fibers/mm^2 filter area
BIAS:	See EVALUATION OF METHOD	**PRECISION ($\hat{S}_r$):**	0.10 to 0.12 [1]; see EVALUATION OF METHOD
OVERALL PRECISION ($\hat{S}_{rT}$):	0.115 to 0.13 [1]		
ACCURACY:	See EVALUATION OF METHOD		

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. The method gives an index of airborne fibers. It is primarily used for estimating asbestos concentrations, though PCM does not differentiate between asbestos and other fibers. Use this method in conjunction with electron microscopy (e.g., Method 7402) for assistance in identification of fibers. Fibers < ca. 0.25 µm diameter will not be detected by this method [4]. This method may be used for other materials such as fibrous glass by using alternate counting rules (see Appendix C).

INTERFERENCES: If the method is used to detect a specific type of fiber, any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chain-like particles may appear fibrous. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

OTHER METHODS: This revision replaces Method 7400, Revision #3 (date 5/15/89).

REAGENTS:

1. Acetone,* reagent grade.
2. Triacetin (glycerol triacetate), reagent grade.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically conductive extension cowl and cellulose ester filter, 0.45- to 1.2-µm pore size, and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use to check for clarity and background. Discard the filter lot if mean is ≥5 fibers per 100 graticule fields. These are defined as laboratory blanks. Manufacturer-provided quality assurance checks on filter blanks are normally adequate as long as field blanks are analyzed as described below.
   NOTE 2: The electrically conductive extension cowl reduces electrostatic effects. Ground the cowl when possible during sampling.
   NOTE 3: Use 0.8-µm pore size filters for personal sampling. The 0.45-µm filters are recommended for sampling when performing TEM analysis on the same samples. However, their higher pressure drop precludes their use with personal sampling pumps.
   NOTE 4: Other cassettes have been proposed that exhibit improved uniformity of fiber deposit on the filter surface, e.g., bellmouthed sampler (Envirometrics, Charleston, SC). These may be used if shown to give measured concentrations equivalent to sampler indicated above for the application.
2. Personal sampling pump, battery or line-powered vacuum, of sufficient capacity to meet flow-rate requirements (see step 4 for flow rate), with flexible connecting tubing.
3. Wire, multi-stranded, 22-gauge; 1", hose clamp to attach wire to cassette.
4. Tape, shrink- or adhesive-.
5. Slides, glass, frosted-end, pre-cleaned, 25 x 75-mm.
6. Cover slips, 22- x 22-mm, No. 1-1/2, unless otherwise specified by microscope manufacturer.
7. Lacquer or nail polish.
8. Knife, #10 surgical steel, curved blade.
9. Tweezers.

EQUIPMENT:

10. Acetone flash vaporization system for clearing filters on glass slides (see ref. [5] for specifications or see manufacturer's instructions for equivalent devices).
11. Micropipets or syringes, 5-µL and 100- to 500-µL.
12. Microscope, positive phase (dark) contrast, with green or blue filter, adjustable field iris, 8 to 10X eyepiece, and 40 to 45X phase objective (total magnification ca. 400X); numerical aperture = 0.65 to 0.75.
13. Graticule, Walton-Beckett type with 100-µm diameter circular field (area = 0.00785 mm2) at the specimen plane (Type G-22). Available from Optometrics USA, P.O. Box 699, Ayer, MA 01432 [phone (508)-772-1700], and McCrone Accessories and Components, 850 Pasquinelli Drive, Westmont, IL 60559 [phone (312) 887-7100].
    NOTE: The graticule is custom-made for each microscope. (see APPENDIX A for the custom-ordering procedure).
14. HSE/NPL phase contrast test slide, Mark II. Available from Optometrics USA (address above).
15. Telescope, ocular phase-ring centering.
16. Stage micrometer (0.01-mm divisions).

SPECIAL PRECAUTIONS: Acetone is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL must be done in a ventilated laboratory fume hood using a flameless, spark- free heat source.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. To reduce contamination and to hold the cassette tightly together, seal the crease between the cassette base and the cowl with a shrink band or light colored adhesive tape. For personal sampling, fasten the (uncapped) open-face cassette to the worker's lapel. The open face should be oriented downward.
   NOTE: The cowl should be electrically grounded during area sampling, especially under conditions of low relative humidity. Use a hose clamp to secure one end of the wire (Equipment, Item 3) to the monitor's cowl. Connect the other end to an earth ground (i.e., cold water pipe).
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.
4. Sample at 0.5 L/min or greater [6]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E, of 100 to 1300 fibers/mm^2 ($3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area A_c= 385 mm^2) for optimum accuracy. These variables are related to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

NOTE 1: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. The collection efficiency does not appear to be a function of flow rate in the range of 0.5 to 16 L/min for asbestos fibers [7]. Relatively large diameter fibers (>3 µm) may exhibit significant aspiration loss and inlet deposition. A sampling rate of 1 to 4 L/min for 8 h is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes (≤400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If ≥ 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

NOTE 2: OSHA regulations specify a minimum sampling volume of 48 L for an excursion measurement, and a maximum sampling rate of 2.5 L/min [3].

5. At the end of sampling, replace top cover and end plugs.
6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.

   NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

NOTE 1: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index ≤1.46. This method collapses the filter for easier focusing and produces permanent (1 - 10 years) mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" or similar flash vaporization techniques may be used outside the laboratory [2]. Other mounting techniques meeting the above criteria may also be used (e.g., the laboratory fume hood procedure for generating acetone vapor as described in Method 7400 - revision of 5/15/85, or the non-permanent field mounting technique used in P&CAM 239 [3,7,8,9]). Unless the effective filtration area is known, determine the area and record the information referenced against the sample ID number [1,9,10,11].

NOTE 2: Excessive water in the acetone may slow the clearing of the filter, causing material to be washed off the surface of the filter. Also, filters that have been exposed to high humidities prior to clearing may have a grainy background.

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Adjust the rheostat to heat the "hot block" to ca. 70 °C [2].

   NOTE: If the "hot block" is not used in a fume hood, it must rest on a ceramic plate and be isolated from any surface susceptible to heat damage.
9. Mount a wedge cut from the sample filter on a clean glass slide.
   a. Cut wedges of ca. 25% of the filter area with a curved-blade surgical steel knife using a rocking motion to prevent tearing. Place wedge, dust side up, on slide.

      NOTE: Static electricity will usually keep the wedge on the slide.

b. Insert slide with wedge into the receiving slot at base of "hot block". Immediately place tip of a micropipet containing ca. 250 µL acetone (use the minimum volume needed to consistently clear the filter sections) into the inlet port of the PTFE cap on top of the "hot block" and inject the acetone into the vaporization chamber with a slow, steady pressure on the plunger button while holding pipet firmly in place. After waiting 3 to 5 sec for the filter to clear, remove pipet and slide from their ports.

CAUTION: Although the volume of acetone used is small, use safety precautions. Work in a well-ventilated area (e.g., laboratory fume hood). Take care not to ignite the acetone. Continuous use of this device in an unventilated space may produce explosive acetone vapor concentrations.

c. Using the 5-µL micropipet, immediately place 3.0 to 3.5 µL triacetin on the wedge. Gently lower a clean cover slip onto the wedge at a slight angle to reduce bubble formation. Avoid excess pressure and movement of the cover glass.

NOTE: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur.

d. Mark the outline of the filter segment with a glass marking pen to aid in microscopic evaluation.

e. Glue the edges of the cover slip to the slide using lacquer or nail polish [12]. Counting may proceed immediately after clearing and mounting are completed.

NOTE: If clearing is slow, warm the slide on a hotplate (surface temperature 50 °C) for up to 15 min to hasten clearing. Heat carefully to prevent gas bubble formation.

CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturers instructions. At least once daily use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a logbook in which to record the dates of microscope cleanings and major servicing.
    a. Each time a sample is examined, do the following:
        (1) Adjust the light source for even illumination across the field of view at the condenser iris. Use Kohler illumination, if available. With some microscopes, the illumination may have to be set up with bright field optics rather than phase contract optics.
        (2) Focus on the particulate material to be examined.
        (3) Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.
    b. Check the phase-shift detection limit of the microscope periodically for each analyst/microscope combination:
        (1) Center the HSE/NPL phase-contrast test slide under the phase objective.
        (2) Bring the blocks of grooved lines into focus in the graticule area.

        NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting the microscope optics must completely resolve the grooved lines in block 3 although they may appear somewhat faint, and the grooved lines in blocks 6 and 7 must be invisible when centered in the graticule area. Blocks 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has resolution either too low or too high for fiber counting.

        (3) If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.
11. Document the laboratory's precision for each counter for replicate fiber counts.
    a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis [13]. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field and reference samples (e.g., PAT, AAR, commercial samples). The Quality Assurance Officer

should maintain custody of the reference slides and should supply each counter with a minimum of one reference slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.

b. From blind repeat counts on reference slides, estimate the laboratory intra- and intercounter precision. Obtain separate values of relative standard deviation (S_r) for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, >20 to 50 fibers in 100 graticule fields, and >50 to 100 fibers in 100 graticule fields. Maintain control charts for each of these data files.

NOTE: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision [9]

12. Prepare and count field blanks along with the field samples. Report counts on each field blank.

NOTE 1: The identity of blank filters should be unknown to the counter until all counts have been completed.

NOTE 2: If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

13. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: Discard the sample if the absolute value of the difference between the square roots of the two counts (in fiber/mm^2) exceeds 2.77 $(X)S_r'$, where X = average of the square roots of the two fiber counts

(in fiber/mm^2) and $S_r' = \frac{S_r}{2}$, where S_r is the intracounter relative standard deviation for the

appropriate count range (in fibers) determined in step 11. For more complete discussions see reference [13].

NOTE 1: Since fiber counting is the measurement of randomly placed fibers which may be described by a Poisson distribution, a square root transformation of the fiber count data will result in approximately normally distributed data [13].

NOTE 2: If a pair of counts is rejected by this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.

14. The analyst is a critical part of this analytical procedure. Care must be taken to provide a non-stressful and comfortable environment for fiber counting. An ergonomically designed chair should be used, with the microscope eyepiece situated at a comfortable height for viewing. External lighting should be set at a level similar to the illumination level in the microscope to reduce eye fatigue. In addition, counters should take 10-to-20 minute breaks from the microscope every one or two hours to limit fatigue [14]. During these breaks, both eye and upper back/neck exercises should be performed to relieve strain.

15. All laboratories engaged in asbestos counting should participate in a proficiency testing program such as the AIHA-NIOSH Proficiency Analytical Testing (PAT) Program for asbestos and routinely exchange field samples with other laboratories to compare performance of counters.

MEASUREMENT:

16. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the plane of the filter.

17. Adjust the microscope (Step 10).

NOTE: Calibration with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25 µm) [4].

18. Counting rules: (same as P&CAM 239 rules [1,10,11]: see examples in APPENDIX B).

a. Count any fiber longer than 5 µm which lies entirely within the graticule area.

(1) Count only fibers longer than 5 µm. Measure length of curved fibers along the curve.

(2) Count only fibers with a length-to-width ratio equal to or greater than 3:1.

b. For fibers which cross the boundary of the graticule field:

(1) Count as 1/2 fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of rule a above.

(2) Do not count any fiber which crosses the graticule boundary more than once.
(3) Reject and do not count all other fibers.

c. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
d. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.

19. Start counting from the tip of the filter wedge and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate or bubble covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not report rejected graticule fields in the total number counted.

NOTE 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 seconds per field is appropriate for accurate counting.

NOTE 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestiform, there is presently no accepted method for ensuring uniformity of judgment between laboratories. It is, therefore, incumbent upon all laboratories using this method to report total fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample (see NIOSH Method 7402). In some cases (i.e., for fibers with diameters >1 µm), polarized light microscopy (as in NIOSH Method 7403) may be used to identify and eliminate interfering non-crystalline fibers [15].

NOTE 3: Do not count at edges where filter was cut. Move in at least 1 mm from the edge.

NOTE 4: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (below 20%), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. If such a pattern is noted during fiber counting, choose fields as close to the center of the filter as possible [5].

NOTE 5: Counts are to be recorded on a data sheet that provides, as a minimum, spaces on which to record the counts for each field, filter identification number, analyst's name, date, total fibers counted, total fields counted, average count, fiber density, and commentary. Average count is calculated by dividing the total fiber count by the number of fields observed. Fiber density (fibers/mm^2) is defined as the average count (fibers/field) divided by the field (graticule) area (mm^2/field).

CALCULATIONS AND REPORTING OF RESULTS

20. Calculate and report fiber density on the filter, E (fibers/mm^2), by dividing the average fiber count per graticule field, F/n_f, minus the mean field blank count per graticule field, B/n_b, by the graticule field area, A_f (approx. 0.00785 mm^2):

$$E = \frac{\left(\frac{F}{n_f} - \frac{B}{n_b}\right)}{A_f}, \text{ fibers/mm}^2.$$

NOTE: Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of filter area covered with particulate should be reported as "uncountable" or "probably biased." Other fiber counts outside the 100-1300 fiber/mm^2 range should be reported as having "greater than optimal variability" and as being "probably biased."

21. Calculate and report the concentration, C (fibers/cc), of fibers in the air volume sampled, V (L), using the effective collection area of the filter, A_c (approx. 385 mm^2 for a 25-mm filter):

$$C = \frac{(E)(A_c)}{V \cdot 10^3}.$$

NOTE: Periodically check and adjust the value of A_c, if necessary.

22. Report intralaboratory and interlaboratory relative standard deviations (from Step 11) with each set of results.

NOTE: Precision depends on the total number of fibers counted [1,16]. Relative standard deviation is documented in references [1,15-17] for fiber counts up to 100 fibers in 100 graticule fields. Comparability of interlaboratory results is discussed below. As a first approximation, use 213% above and 49% below the count as the upper and lower confidence limits for fiber counts greater than 20 (Fig. 1).

EVALUATION OF METHOD:

A. This method is a revision of P&CAM 239 [10]. A summary of the revisions is as follows:

1. Sampling:
   The change from a 37-mm to a 25-mm filter improves sensitivity for similar air volumes. The change in flow rates allows for 2-m^3 full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not a function of flow rate in the range 0.5 to 16 L/min [10].
2. Sample Preparation Technique:
   The acetone vapor-triacetin preparation technique is a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [2,4,10]. The aluminum "hot block" technique minimizes the amount of acetone needed to prepare each sample.
3. Measurement:
   a. The Walton-Beckett graticule standardizes the area observed [14,18,19].
   b. The HSE/NPL test slide standardizes microscope optics for sensitivity to fiber diameter [4,14].
   c. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading has been increased to 100 fibers/mm^2 filter area (a total of 78.5 fibers counted in 100 fields, each with field area = .00785 mm^2.) Lower levels generally result in an overestimate of the fiber count when compared to results in the recommended analytical range [20]. The recommended loadings should yield intracounter S_r in the range of 0.10 to 0.17 [21,22,23].

B. Interlaboratory comparability:
An international collaborative study involved 16 laboratories using prepared slides from the asbestos cement, milling, mining, textile, and friction material industries [9]. The relative standard deviations (S_r) varied with sample type and laboratory. The ranges were:

	Intralaboratory S_r	Interlaboratory S_r	Overall S_r
AIA (NIOSH A Rules)*	0.12 to 0.40	0.27 to 0.85	0.46
Modified CRS (NIOSH B Rules)**	0.11 to 0.29	0.20 to 0.35	0.25

* Under AIA rules, only fibers having a diameter less than 3 µm are counted and fibers attached to particles larger than 3 µm are not counted. NIOSH A Rules are otherwise similar to the AIA rules.
** See Appendix C.

A NIOSH study conducted using field samples of asbestos gave intralaboratory S_r in the range 0.17 to 0.25 and an interlaboratory S_r of 0.45 [21]. This agrees well with other recent studies [9,14,16].

At this time, there is no independent means for assessing the overall accuracy of this method. One measure of reliability is to estimate how well the count for a single sample agrees with the mean count from a large number of laboratories. The following discussion indicates how this estimation can be carried out based on measurements of the interlaboratory variability, as well as showing how the results of this method relate to the theoretically attainable counting precision and to measured intra- and interlaboratory S_r. (NOTE: The following discussion does not include bias estimates and should not be taken to indicated that lightly loaded samples are as accurate as properly loaded ones).

Theoretically, the process of counting randomly (Poisson) distributed fibers on a filter surface will give an S_r that depends on the number, N, of fibers counted:

$$S_r = 1/(N)^{1/2} \tag{1}$$

Thus S_r is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual S_r found in a number of studies is greater than these theoretical numbers [17,19,20,21].

An additional component of variability comes primarily from subjective interlaboratory differences. In a study of ten counters in a continuing sample exchange program, Ogden [15] found this subjective component of intralaboratory S_r to be approximately 0.2 and estimated the overall S_r by the term:

$$\frac{[N + (0.2 \cdot N)^2]^{1/2}}{N} \tag{2}$$

Ogden found that the 90% confidence interval of the individual intralaboratory counts in relation to the means were +2 S_r and - 1.5 S_r. In this program, one sample out of ten was a quality control sample. For laboratories not engaged in an intensive quality assurance program, the subjective component of variability can be higher.

In a study of field sample results in 46 laboratories, the Asbestos Information Association also found that the variability had both a constant component and one that depended on the fiber count [14]. These results gave a subjective interlaboratory component of S_r (on the same basis as Ogden's) for field samples of ca. 0.45. A similar value was obtained for 12 laboratories analyzing a set of 24 field samples [21]. This value falls slightly above the range of S_r (0.25 to 0.42 for 1984-85) found for 80 reference laboratories in the NIOSH PAT program for laboratory-generated samples [17].

A number of factors influence S_r for a given laboratory, such as that laboratory's actual counting performance and the type of samples being analyzed. In the absence of other information, such as from an interlaboratory quality assurance program using field samples, the value for the subjective component of variability is chosen as 0.45. It is hoped that the laboratories will carry out the recommended interlaboratory quality assurance programs to improve their performance and thus reduce the S_r.

The above relative standard deviations apply when the population mean has been determined. It is more useful, however, for laboratories to estimate the 90% confidence interval on the mean count from a single sample fiber count (Figure 1). These curves assume similar shapes of the count distribution for interlaboratory and intralaboratory results [16].

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. We can apply these percentages directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/mL (assuming 100 fields counted, 25-mm filter, 0.00785 mm^2 counting field area). If this same sample were counted by a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/mL. These limits should be reported in any comparison of results between laboratories.

Note that the S_r of 0.45 used to derive Figure 1 is used as an estimate for a random group of laboratories. If several laboratories belonging to a quality assurance group can show that their interlaboratory S_r is smaller, then it is more correct to use that smaller S_r. However, the estimated S_r of 0.45 is to be used in the absence of such information. Note also that it has been found that S_r can be higher for certain types of samples, such as asbestos cement [9].

Quite often the estimated airborne concentration from an asbestos analysis is used to compare to a regulatory standard. For instance, if one is trying to show compliance with an 0.5 fiber/mL standard using a single sample on which 100 fibers have been counted, then Figure 1 indicates that the 0.5 fiber/mL standard must be 213% higher than the measured air concentration. This indicates that if one measures a fiber concentration of 0.16 fiber/mL (100 fibers counted), then the mean fiber count by a group of laboratories (of which the compliance laboratory might be one) has a 95% chance of being less than 0.5 fibers/mL; i.e., 0.16 + 2.13 x 0.16 = 0.5.

It can be seen from Figure 1 that the Poisson component of the variability is not very important unless the number of fibers counted is small. Therefore, a further approximation is to simply use +213% and −49% as the upper and lower confidence values of the mean for a 100-fiber count.

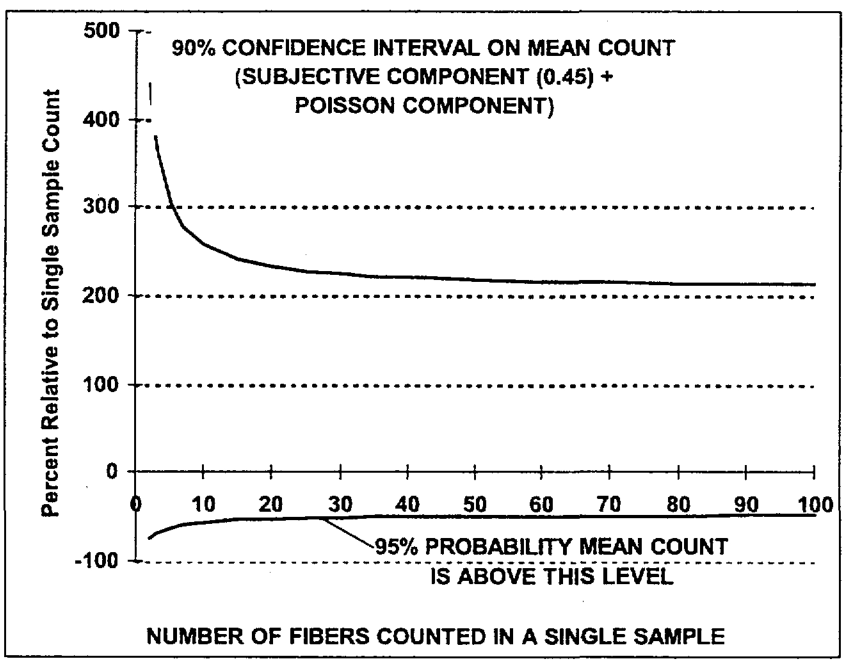


Figure 1. Interlaboratory Precision of Fiber Counts

The curves in Figures 1 are defined by the following equations:

$$\mathbf{UCL} = \frac{2X + 2.25 + [(2.25 + 2X)^2 - 4(1 - 2.25 S_r^2) X^2]^{1/2}}{2(1 - 2.25 S_r^2)} \tag{3}$$

$$\mathbf{LCL} = \frac{2X + 4 - [(4 + 2X)^2 - 4(1 - 4 S_r^2) X^2]^{1/2}}{2(1 - 4 S_r^2)} \tag{4}$$

where S_r = subjective interlaboratory relative standard deviation, which is close to the total interlaboratory S_r when approximately 100 fibers are counted.
X = total fibers counted on sample
LCL = lower 95% confidence limit.
UCL = upper 95% confidence limit.
Note that the range between these two limits represents 90% of the total range.

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APPENDIX A: CALIBRATION OF THE WALTON-BECKETT GRATICULE:

Before ordering the Walton-Beckett graticule, the following calibration must be done to obtain a counting area (D) 100 µm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

1. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
2. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
3. Install the 40 to 45X phase objective.
4. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
5. Measure the magnified grid length of the graticule, L_o (µm), using the stage micrometer.
6. Remove the graticule from the microscope and measure its actual grid length, L_a (mm). This can best be accomplished by using a stage fitted with verniers.
7. Calculate the circle diameter, d_c (mm), for the Walton-Beckett graticule:

$$d_c = \frac{L_a}{L_o} \times D. \qquad (5)$$

Example: If L_o = 112 µm, L_a = 4.5 mm and D = 100 µm, then d_c = 4.02 mm.

8. Check the field diameter, D (acceptable range 100 µm ± 2 µm) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (acceptable range 0.00754 mm^2 to 0.00817 mm^2).

APPENDIX B: COMPARISON OF COUNTING RULES:

Figure 2 shows a Walton-Beckett graticule as seen through the microscope. The rules will be discussed as they apply to the labeled objects in the figure.

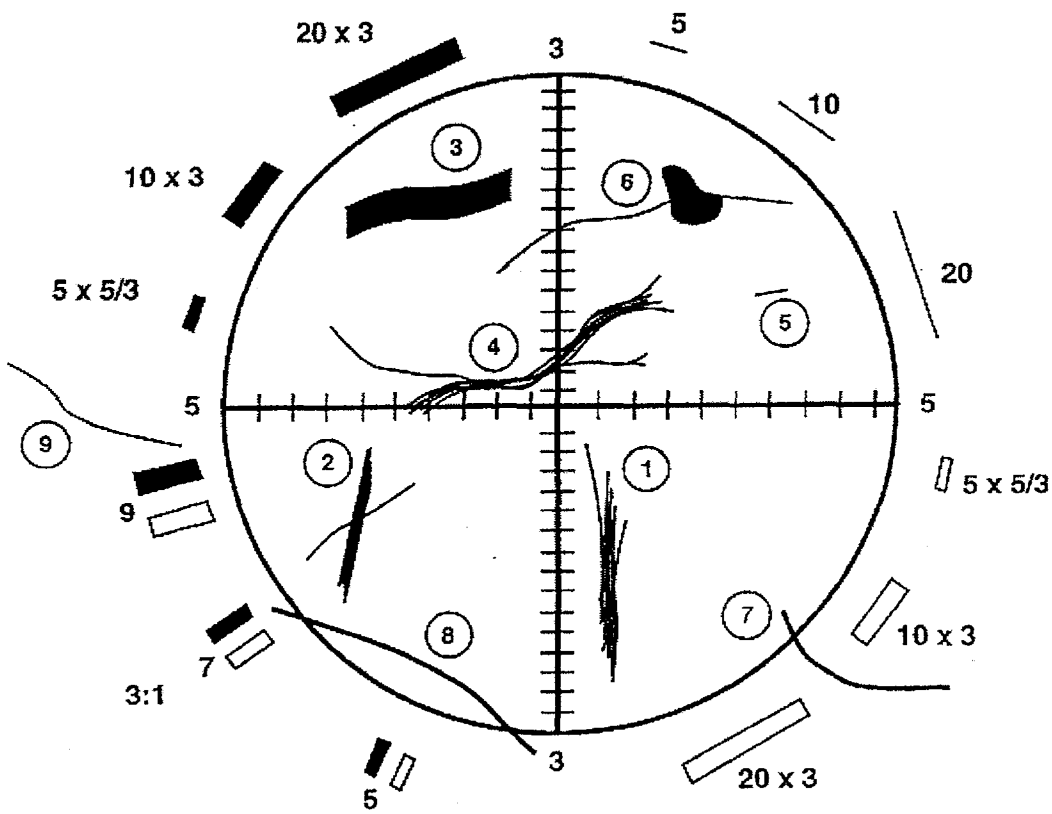

Figure 2. Walton-Beckett graticule with fibers.

These rules are sometimes referred to as the "A" rules.

FIBER COUNT

Object	Count	DISCUSSION
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fiber	If fibers meeting the length and aspect ratio criteria (length >5 µm and length-to-width ratio >3 to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter (>3 µm), it is counted as fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is ≤5 µm long, it is not counted.
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	1/2 fiber	A fiber which crosses into the graticule area one time is counted as 1/2 fiber.
8	Do not count	Ignore fibers that cross the graticulate boundary more than once. count
9	Do not count	Ignore fibers that lie outside the graticule boundary.

APPENDIX C. ALTERNATE COUNTING RULES FOR NON-ASBESTOS FIBERS

Other counting rules may be more appropriate for measurement of specific non-asbestos fiber types, such as fibrous glass. These include the "B" rules given below (from NIOSH Method 7400, Revision #2, dated 8/15/87), the World Health Organization reference method for man-made mineral fiber [24], and the NIOSH fibrous glass criteria document method [25]. The upper diameter limit in these methods prevents measurements of non-thoracic fibers. It is important to note that the aspect ratio limits included in these methods vary. NIOSH recommends the use of the 3:1 aspect ratio in counting fibers.

It is emphasized that hybridization of different sets of counting rules is not permitted. Report specifically which set of counting rules are used with the analytical results.

"B" COUNTING RULES:

1. Count only <u>ends</u> of fibers. Each fiber must be longer than 5 µm and less than 3 µm diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules 1 and 2 above. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of rules 1 and 2 above.
4. Count visibly free ends which meet rules 1 and 2 above when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3 µm in diameter.
5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules 1 and 2 above.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 graticule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

APPENDIX D. EQUIVALENT LIMITS OF DETECTION AND QUANTITATION

	fiber density on filter*		fiber concentration in air, f/cc	
	fibers per 100 fields	fibers/mm^2	400-L air sample	1000-L air sample
	200	255	0.25	0.10
	100	127	0.125	0.05
LOQ	80	102	0.10	0.04
	50	64	0.0625	0.025
	25	32	0.03	0.0125
	20	25	0.025	0.010
	10	12.7	0.0125	0.005
	8	10.2	0.010	0.004
LOD	5.5	7	0.00675	0.0027

* Assumes 385 mm^2 effective filter collection area, and field area = 0.00785 mm^2, for relatively "clean" (little particulate aside from fibers) filters.

contaminated surface used in the validation study. Record and keep the results of the validation study as an appendix to the SOP. Include in this appendix, the solvent used to make the spiking solution, the PCB concentration of the spiking solution used to contaminate the surfaces in the validation study, and all of the validation study testing parameters and experimental conditions.

PART 763—ASBESTOS

Subparts A–C [Reserved]

Subpart D [Reserved]

Subpart E—Asbestos-Containing Materials in Schools

Subpart F [Reserved]

Subpart G—Asbestos Abatement Projects

Subpart H [Reserved]

Subpart I—Prohibition of the Manufacture, Importation, Processing, and Distribution in Commerce of Certain Asbestos-Containing Products; Labeling Requirements

AUTHORITY: 15 U.S.C. 2605, 2607(c), 2643, and 2646.

Subparts A–C [Reserved]

Subpart D [Reserved]

Subpart E—Asbestos-Containing Materials in Schools

SOURCE: 52 FR 41846, Oct. 30, 1987, unless otherwise noted.

§ 763.80 Scope and purpose.

(a) This rule requires local education agencies to identify friable and nonfriable asbestos-containing material (ACM) in public and private elementary and secondary schools by visually inspecting school buildings for such materials, sampling such materials if they are not assumed to be ACM, and having samples analyzed by appropriate techniques referred to in this rule. The rule requires local education agencies to submit management plans to the Governor of their State by October 12, 1988, begin to implement the plans by July 9, 1989, and complete implementation of the plans in a timely fashion. In addition, local education agencies are required to use persons who have been accredited to conduct inspections, reinspections, develop management plans, or perform response actions. The rule also includes recordkeeping requirements. Local education agencies may contractually delegate their duties under this rule, but they remain responsible for the proper performance of those duties. Local education agencies are encouraged to consult with EPA Regional Asbestos Coordinators, or if applicable, a State's lead agency designated by the State Governor, for assistance in complying with this rule.

(b) Local education agencies must provide for the transportation and disposal of asbestos in accordance with EPA's "Asbestos Waste Management Guidance." For convenience, applicable sections of this guidance are reprinted as Appendix D of this subpart. There are regulations in place, however, that affect transportation and disposal of asbestos waste generated by this rule. The transportation of asbestos waste is covered by the Department of Transportation (49 CFR part 173, subpart J) and disposal is covered by the National Emissions Standards for Hazardous Air Pollutants (NESHAP) (40 CFR part 61, subpart M).

§ 763.83 Definitions.

For purposes of this subpart:

Act means the Toxic Substances Control Act (TSCA), 15 U.S.C. 2601, *et seq.*

Accessible when referring to ACM means that the material is subject to disturbance by school building occupants or custodial or maintenance personnel in the course of their normal activities.

Accredited or *accreditation* when referring to a person or laboratory means that such person or laboratory is accredited in accordance with section 206 of Title II of the Act.

Air erosion means the passage of air over friable ACBM which may result in the release of asbestos fibers.

Asbestos means the asbestiform varieties of: Chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonitegrunerite); anthophyllite; tremolite; and actinolite.

Asbestos-containing material (ACM) when referring to school buildings means any material or product which contains more than 1 percent asbestos.

Asbestos-containing building material (ACBM) means surfacing ACM, thermal system insulation ACM, or miscellaneous ACM that is found in or on interior structural members or other parts of a school building.

Asbestos debris means pieces of ACBM that can be identified by color, texture, or composition, or means dust, if the dust is determined by an accredited inspector to be ACM.

Damaged friable miscellaneous ACM means friable miscellaneous ACM which has deteriorated or sustained physical injury such that the internal structure (cohesion) of the material is inadequate or, if applicable, which has delaminated such that its bond to the substrate (adhesion) is inadequate or which for any other reason lacks fiber cohesion or adhesion qualities. Such damage or deterioration may be illustrated by the separation of ACM into layers; separation of ACM from the substrate; flaking, blistering, or crumbling of the ACM surface; water damage; significant or repeated water stains, scrapes, gouges, mars or other signs of physical injury on the ACM. Asbestos debris originating from the ACBM in question may also indicate damage.

Damaged friable surfacing ACM means friable surfacing ACM which has deteriorated or sustained physical injury such that the internal structure (cohesion) of the material is inadequate or which has delaminated such that its bond to the substrate (adhesion) is inadequate, or which, for any other reason, lacks fiber cohesion or adhesion qualities. Such damage or deterioration may be illustrated by the separation of ACM into layers; separation of ACM from the substrate; flaking, blistering, or crumbling of the ACM surface; water damage; significant or repeated water stains, scrapes, gouges, mars or other signs of physical injury on the ACM. Asbestos debris originating from the ACBM in question may also indicate damage.

Damaged or significantly damaged thermal system insulation ACM means thermal system insulation ACM on pipes, boilers, tanks, ducts, and other thermal system insulation equipment where the insulation has lost its structural integrity, or its covering, in whole or in part, is crushed, water-stained, gouged, punctured, missing, or not intact such that it is not able to contain fibers. Damage may be further illustrated by occasional punctures, gouges or other signs of physical injury to ACM; occasional water damage on the protective coverings/jackets; or exposed ACM ends or joints. Asbestos debris originating from the ACBM in question may also indicate damage.

Encapsulation means the treatment of ACBM with a material that surrounds or embeds asbestos fibers in an adhesive matrix to prevent the release of fibers, as the encapsulant creates a membrane over the surface (bridging encapsulant) or penetrates the material and binds its components together (penetrating encapsulant).

Enclosure means an airtight, impermeable, permanent barrier around ACBM to prevent the release of asbestos fibers into the air.

Fiber release episode means any uncontrolled or unintentional disturbance of ACBM resulting in visible emission.

Friable when referring to material in a school building means that the material, when dry, may be crumbled, pulverized, or reduced to powder by hand pressure, and includes previously nonfriable material after such previously nonfriable material becomes damaged to the extent that when dry it may be crumbled, pulverized, or reduced to powder by hand pressure.

Functional space means a room, group of rooms, or homogeneous area (including crawl spaces or the space between a dropped ceiling and the floor or roof deck above), such as classroom(s), a cafeteria, gymnasium, hallway(s), designated by a person accredited to prepare management plans, design abatement projects, or conduct response actions.

High-efficiency particulate air (HEPA) refers to a filtering system capable of trapping and retaining at least 99.97 percent of all monodispersed particles 0.3 μm in diameter or larger.

Homogeneous area means an area of surfacing material, thermal system insulation material, or miscellaneous material that is uniform in color and texture.

Local education agency means:

(1) Any local educational agency as defined in section 198 of the Elementary and Secondary Education Act of 1965 (20 U.S.C. 3381).

(2) The owner of any nonpublic, nonprofit elementary, or secondary school building.

(3) The governing authority of any school operated under the defense dependent's education system provided for under the Defense Dependents' Education Act of 1978 (20 U.S.C. 921, et seq.).

Miscellaneous ACM means miscellaneous material that is ACM in a school building.

Miscellaneous material means interior building material on structural components, structural members or fixtures, such as floor and ceiling tiles, and does not include surfacing material or thermal system insulation.

Nonfriable means material in a school building which when dry may not be crumbled, pulverized, or reduced to powder by hand pressure.

Operations and maintenance program means a program of work practices to maintain friable ACBM in good condition, ensure clean up of asbestos fibers previously released, and prevent further release by minimizing and controlling friable ACBM disturbance or damage.

Potential damage means circumstances in which:

(1) Friable ACBM is in an area regularly used by building occupants, including maintenance personnel, in the course of their normal activities.

(2) There are indications that there is a reasonable likelihood that the material or its covering will become damaged, deteriorated, or delaminated due to factors such as changes in building use, changes in operations and maintenance practices, changes in occupancy, or recurrent damage.

Potential significant damage means circumstances in which:

(1) Friable ACBM is in an area regularly used by building occupants, including maintenance personnel, in the course of their normal activities.

(2) There are indications that there is a reasonable likelihood that the material or its covering will become significantly damaged, deteriorated, or delaminated due to factors such as

changes in building use, changes in operations and maintenance practices, changes in occupancy, or recurrent damage.

(3) The material is subject to major or continuing disturbance, due to factors including, but not limited to, accessibility or, under certain circumstances, vibration or air erosion.

Preventive measures means actions taken to reduce disturbance of ACBM or otherwise eliminate the reasonable likelihood of the material's becoming damaged or significantly damaged.

Removal means the taking out or the stripping of substantially all ACBM from a damaged area, a functional space, or a homogeneous area in a school building.

Repair means returning damaged ACBM to an undamaged condition or to an intact state so as to prevent fiber release.

Response action means a method, including removal, encapsulation, enclosure, repair, operations and maintenance, that protects human health and the environment from friable ACBM.

Routine maintenance area means an area, such as a boiler room or mechanical room, that is not normally frequented by students and in which maintenance employees or contract workers regularly conduct maintenance activities.

School means any elementary or secondary school as defined in section 198 of the Elementary and Secondary Education Act of 1965 (20 U.S.C. 2854).

School building means:

(1) Any structure suitable for use as a classroom, including a school facility such as a laboratory, library, school eating facility, or facility used for the preparation of food.

(2) Any gymnasium or other facility which is specially designed for athletic or recreational activities for an academic course in physical education.

(3) Any other facility used for the instruction or housing of students or for the administration of educational or research programs.

(4) Any maintenance, storage, or utility facility, including any hallway, essential to the operation of any facility described in this definition of "school building" under paragraphs (1), (2), or (3).

(5) Any portico or covered exterior hallway or walkway.

(6) Any exterior portion of a mechanical system used to condition interior space.

Significantly damaged friable miscellaneous ACM means damaged friable miscellaneous ACM where the damage is extensive and severe.

Significantly damaged friable surfacing ACM means damaged friable surfacing ACM in a functional space where the damage is extensive and severe.

State means a State, the District of Columbia, the Commonwealth of Puerto Rico, Guam, American Samoa, the Northern Marianas, the Trust Territory of the Pacific Islands, and the Virgin Islands.

Surfacing ACM means surfacing material that is ACM.

Surfacing material means material in a school building that is sprayed-on, troweled-on, or otherwise applied to surfaces, such as acoustical plaster on ceilings and fireproofing materials on structural members, or other materials on surfaces for acoustical, fireproofing, or other purposes.

Thermal system insulation means material in a school building applied to pipes, fittings, boilers, breeching, tanks, ducts, or other interior structural components to prevent heat loss or gain, or water condensation, or for other purposes.

Thermal system insulation ACM means thermal system insulation that is ACM.

Vibration means the periodic motion of friable ACBM which may result in the release of asbestos fibers.

§ 763.84 General local education agency responsibilities.

Each local education agency shall:

(a) Ensure that the activities of any persons who perform inspections, reinspections, and periodic surveillance, develop and update management plans, and develop and implement response actions, including operations and maintenance, are carried out in accordance with subpart E of this part.

(b) Ensure that all custodial and maintenance employees are properly trained as required by this subpart E and other applicable Federal and/or

State regulations (e.g., the Occupational Safety and Health Administration asbestos standard for construction, the EPA worker protection rule, or applicable State regulations).

(c) Ensure that workers and building occupants, or their legal guardians, are informed at least once each school year about inspections, response actions, and post-response action activities, including periodic reinspection and surveillance activities that are planned or in progress.

(d) Ensure that short-term workers (e.g., telephone repair workers, utility workers, or exterminators) who may come in contact with asbestos in a school are provided information regarding the locations of ACBM and suspected ACBM assumed to be ACM.

(e) Ensure that warning labels are posted in accordance with § 763.95.

(f) Ensure that management plans are available for inspection and notification of such availability has been provided as specified in the management plan under § 763.93(g).

(g)(1) Designate a person to ensure that requirements under this section are properly implemented.

(2) Ensure that the designated person receives adequate training to perform duties assigned under this section. Such training shall provide, as necessary, basic knowledge of:

(i) Health effects of asbestos.

(ii) Detection, identification, and assessment of ACM.

(iii) Options for controlling ACBM.

(iv) Asbestos management programs.

(v) Relevant Federal and State regulations concerning asbestos, including those in this subpart E and those of the Occupational Safety and Health Administration, U.S. Department of Labor, the U.S. Department of Transportation and the U.S. Environmental Protection Agency.

(h) Consider whether any conflict of interest may arise from the interrelationship among accredited personnel and whether that should influence the selection of accredited personnel to perform activities under this subpart.

§ 763.85 Inspection and reinspections.

(a) *Inspection.* (1) Except as provided in paragraph (a)(2) of this section, before October 12, 1988, local education agencies shall inspect each school building that they lease, own, or otherwise use as a school building to identify all locations of friable and nonfriable ACBM.

(2) Any building leased or acquired on or after October 12, 1988, that is to be used as a school building shall be inspected as described under paragraphs (a) (3) and (4) of this section prior to use as a school building. In the event that emergency use of an uninspected building as a school building is necessitated, such buildings shall be inspected within 30 days after commencement of such use.

(3) Each inspection shall be made by an accredited inspector.

(4) For each area of a school building, except as excluded under § 763.99, each person performing an inspection shall:

(i) Visually inspect the area to identify the locations of all suspected ACBM.

(ii) Touch all suspected ACBM to determine whether they are friable.

(iii) Identify all homogeneous areas of friable suspected ACBM and all homogeneous areas of nonfriable suspected ACBM.

(iv) Assume that some or all of the homogeneous areas are ACM, and, for each homogeneous area that is not assumed to be ACM, collect and submit for analysis bulk samples under §§ 763.86 and 763.87.

(v) Assess, under § 763.88, friable material in areas where samples are collected, friable material in areas that are assumed to be ACBM, and friable ACBM identified during a previous inspection.

(vi) Record the following and submit to the person designated under § 763.84 a copy of such record for inclusion in the management plan within 30 days of the inspection:

(A) An inspection report with the date of the inspection signed by each accredited person making the inspection, State of accreditation, and if applicable, his or her accreditation number.

(B) An inventory of the locations of the homogeneous areas where samples are collected, exact location where each bulk sample is collected, dates that samples are collected, homogeneous areas where friable suspected ACBM is assumed to be ACM, and homogeneous areas where nonfriable suspected ACBM is assumed to be ACM.

(C) A description of the manner used to determine sampling locations, the name and signature of each accredited inspector who collected the samples, State of accreditation, and, if applicable, his or her accreditation number.

(D) A list of whether the homogeneous areas identified under paragraph (a)(4)(vi)(B) of this section, are surfacing material, thermal system insulation, or miscellaneous material.

(E) Assessments made of friable material, the name and signature of each accredited inspector making the assessment, State of accreditation, and if applicable, his or her accreditation number.

(b) *Reinspection.* (1) At least once every 3 years after a management plan is in effect, each local education agency shall conduct a reinspection of all friable and nonfriable known or assumed ACBM in each school building that they lease, own, or otherwise use as a school building.

(2) Each inspection shall be made by an accredited inspector.

(3) For each area of a school building, each person performing a reinspection shall:

(i) Visually reinspect, and reassess, under § 763.88, the condition of all friable known or assumed ACBM.

(ii) Visually inspect material that was previously considered nonfriable ACBM and touch the material to determine whether it has become friable since the last inspection or reinspection.

(iii) Identify any homogeneous areas with material that has become friable since the last inspection or reinspection.

(iv) For each homogeneous area of newly friable material that is already assumed to be ACBM, bulk samples may be collected and submitted for analysis in accordance with §§ 763.86 and 763.87.

(v) Assess, under § 763.88, the condition of the newly friable material in areas where samples are collected, and newly friable materials in areas that are assumed to be ACBM.

(vi) Reassess, under § 763.88, the condition of friable known or assumed ACBM previously identified.

(vii) Record the following and submit to the person designated under § 763.84 a copy of such record for inclusion in the management plan within 30 days of the reinspection:

(A) The date of the reinspection, the name and signature of the person making the reinspection, State of accreditation, and if applicable, his or her accreditation number, and any changes in the condition of known or assumed ACBM.

(B) The exact locations where samples are collected during the reinspection, a description of the manner used to determine sampling locations, the name and signature of each accredited inspector who collected the samples, State of accreditation, and, if applicable, his or her accreditation number.

(C) Any assessments or reassessments made of friable material, the name and signature of the accredited inspector making the assessments, State of accreditation, and if applicable, his or her accreditation number.

(c) *General.* Thermal system insulation that has retained its structural integrity and that has an undamaged protective jacket or wrap that prevents fiber release shall be treated as nonfriable and therefore is subject only to periodic surveillance and preventive measures as necessary.

§ 763.86 Sampling.

(a) *Surfacing material.* An accredited inspector shall collect, in a statistically random manner that is representative of the homogeneous area, bulk samples from each homogeneous area of friable surfacing material that is not assumed to be ACM, and shall collect the samples as follows:

(1) At least three bulk samples shall be collected from each homogeneous area that is 1,000 ft^2 or less, except as provided in § 763.87(c)(2).

(2) At least five bulk samples shall be collected from each homogeneous area that is greater than 1,000 ft^2 but less than or equal to 5,000 ft^2, except as provided in § 763.87(c)(2).

(3) At least seven bulk samples shall be collected from each homogeneous

area that is greater than 5,000 ft^2, except as provided in § 763.87(c)(2).

(b) *Thermal system insulation.* (1) Except as provided in paragraphs (b) (2) through (4) of this section and § 763.87(c), an accredited inspector shall collect, in a randomly distributed manner, at least three bulk samples from each homogeneous area of thermal system insulation that is not assumed to be ACM.

(2) Collect at least one bulk sample from each homogeneous area of patched thermal system insulation that is not assumed to be ACM if the patched section is less than 6 linear or square feet.

(3) In a manner sufficient to determine whether the material is ACM or not ACM, collect bulk samples from each insulated mechanical system that is not assumed to be ACM where cement or plaster is used on fittings such as tees, elbows, or valves, except as provided under § 763.87(c)(2).

(4) Bulk samples are not required to be collected from any homogeneous area where the accredited inspector has determined that the thermal system insulation is fiberglass, foam glass, rubber, or other non-ACBM.

(c) *Miscellaneous material.* In a manner sufficient to determine whether material is ACM or not ACM, an accredited inspector shall collect bulk samples from each homogeneous area of friable miscellaneous material that is not assumed to be ACM.

(d) *Nonfriable suspected ACBM.* If any homogeneous area of nonfriable suspected ACBM is not assumed to be ACM, then an accredited inspector shall collect, in a manner sufficient to determine whether the material is ACM or not ACM, bulk samples from the homogeneous area of nonfriable suspected ACBM that is not assumed to be ACM.

§ 763.87 Analysis.

(a) Local education agencies shall have bulk samples, collected under § 763.86 and submitted for analysis, analyzed for asbestos using laboratories accredited by the National Bureau of Standards (NBS). Local education agencies shall use laboratories which have received interim accreditation for polarized light microscopy (PLM) analysis under the EPA Interim Asbestos Bulk Sample Analysis Quality Assurance Program until the NBS PLM laboratory accreditation program for PLM is operational.

(b) Bulk samples shall not be composited for analysis and shall be analyzed for asbestos content by PLM, using the "Interim Method for the Determination of Asbestos in Bulk Insulation Samples" found at appendix E to subpart E of this part.

(c)(1) A homogeneous area is considered not to contain ACM only if the results of all samples required to be collected from the area show asbestos in amounts of 1 percent or less.

(2) A homogeneous area shall be determined to contain ACM based on a finding that the results of at least one sample collected from that area shows that asbestos is present in an amount greater than 1 percent.

(d) The name and address of each laboratory performing an analysis, the date of analysis, and the name and signature of the person performing the analysis shall be submitted to the person designated under § 763.84 for inclusion into the management plan within 30 days of the analysis.

[52 FR 41846, Oct. 30, 1987, as amended at 60 FR 31922, June 19, 1995]

§ 763.88 Assessment.

(a)(1) For each inspection and reinspection conducted under § 763.85 (a) and (c) and previous inspections specified under § 763.99, the local education agency shall have an accredited inspector provide a written assessment of all friable known or assumed ACBM in the school building.

(2) Each accredited inspector providing a written assessment shall sign and date the assessment, provide his or her State of accreditation, and if applicable, accreditation number, and submit a copy of the assessment to the person designated under § 763.84 for inclusion in the management plan within 30 days of the assessment.

(b) The inspector shall classify and give reasons in the written assessment for classifying the ACBM and suspected ACBM assumed to be ACM in the school building into one of the following categories:

(1) Damaged or significantly damaged thermal system insulation ACM.

(2) Damaged friable surfacing ACM.

(3) Significantly damaged friable surfacing ACM.

(4) Damaged or significantly damaged friable miscellaneous ACM.

(5) ACBM with potential for damage.

(6) ACBM with potential for significant damage.

(7) Any remaining friable ACBM or friable suspected ACBM.

(c) Assessment may include the following considerations:

(1) Location and the amount of the material, both in total quantity and as a percentage of the functional space.

(2) Condition of the material, specifying:

(i) Type of damage or significant damage (e.g., flaking, blistering, water damage, or other signs of physical damage).

(ii) Severity of damage (e.g., major flaking, severely torn jackets, as opposed to occasional flaking, minor tears to jackets).

(iii) Extent or spread of damage over large areas or large percentages of the homogeneous area.

(3) Whether the material is accessible.

(4) The material's potential for disturbance.

(5) Known or suspected causes of damage or significant damage (e.g., air erosion, vandalism, vibration, water).

(6) Preventive measures which might eliminate the reasonable likelihood of undamaged ACM from becoming significantly damaged.

(d) The local education agency shall select a person accredited to develop management plans to review the results of each inspection, reinspection, and assessment for the school building and to conduct any other necessary activities in order to recommend in writing to the local education agency appropriate response actions. The accredited person shall sign and date the recommendation, provide his or her State of accreditation, and, if applicable, provide his or her accreditation number, and submit a copy of the recommendation to the person designated under § 763.84 for inclusion in the management plan.

§ 763.90 Response actions.

(a) The local education agency shall select and implement in a timely manner the appropriate response actions in this section consistent with the assessment conducted in § 763.88. The response actions selected shall be sufficient to protect human health and the environment. The local education agency may then select, from the response actions which protect human health and the environment, that action which is the least burdensome method. Nothing in this section shall be construed to prohibit removal of ACBM from a school building at any time, should removal be the preferred response action of the local education agency.

(b) If damaged or significantly damaged thermal system insulation ACM is present in a building, the local education agency shall:

(1) At least repair the damaged area.

(2) Remove the damaged material if it is not feasible, due to technological factors, to repair the damage.

(3) Maintain all thermal system insulation ACM and its covering in an intact state and undamaged condition.

(c)(1) If damaged friable surfacing ACM or damaged friable miscellaneous ACM is present in a building, the local education agency shall select from among the following response actions: encapsulation, enclosure, removal, or repair of the damaged material.

(2) In selecting the response action from among those which meet the definitional standards in § 763.83, the local education agency shall determine which of these response actions protects human health and the environment. For purposes of determining which of these response actions are the least burdensome, the local education agency may then consider local circumstances, including occupancy and use patterns within the school building, and its economic concerns, including short- and long-term costs.

(d) If significantly damaged friable surfacing ACM or significantly damaged friable miscellaneous ACM is present in a building the local education agency shall:

(1) Immediately isolate the functional space and restrict access, unless

isolation is not necessary to protect human health and the environment.

(2) Remove the material in the functional space or, depending upon whether enclosure or encapsulation would be sufficient to protect human health and the environment, enclose or encapsulate.

(e) If any friable surfacing ACM, thermal system insulation ACM, or friable miscellaneous ACM that has potential for damage is present in a building, the local education agency shall at least implement an operations and maintenance (O&M) program, as described under §763.91.

(f) If any friable surfacing ACM, thermal system insulation ACM, or friable miscellaneous ACM that has potential for significant damage is present in a building, the local education agency shall:

(1) Implement an O&M program, as described under §763.91.

(2) Institute preventive measures appropriate to eliminate the reasonable likelihood that the ACM or its covering will become significantly damaged, deteriorated, or delaminated.

(3) Remove the material as soon as possible if appropriate preventive measures cannot be effectively implemented, or unless other response actions are determined to protect human health and the environment. Immediately isolate the area and restrict access if necessary to avoid an imminent and substantial endangerment to human health or the environment.

(g) Response actions including removal, encapsulation, enclosure, or repair, other than small-scale, short-duration repairs, shall be designed and conducted by persons accredited to design and conduct response actions.

(h) The requirements of this subpart E in no way supersede the worker protection and work practice requirements under 29 CFR 1926.58 (Occupational Safety and Health Administration (OSHA) asbestos worker protection standards for construction), 40 CFR part 763, subpart G (EPA asbestos worker protection standards for public employees), and 40 CFR part 61, subpart M (National Emission Standards for Hazardous Air Pollutants—Asbestos).

(i) Completion of response actions. (1) At the conclusion of any action to remove, encapsulate, or enclose ACBM or material assumed to be ACBM, a person designated by the local education agency shall visually inspect each functional space where such action was conducted to determine whether the action has been properly completed.

(2)(i) A person designated by the local education agency shall collect air samples using aggressive sampling as described in appendix A to this subpart E to monitor air for clearance after each removal, encapsulation, and enclosure project involving ACBM, except for projects that are of small-scale, short-duration.

(ii) Local education agencies shall have air samples collected under this section analyzed for asbestos using laboratories accredited by the National Bureau of Standards to conduct such analysis using transmission electron microscopy (TEM) or, under circumstances permitted in this section, laboratories enrolled in the American Industrial Hygiene Association Proficiency Analytical Testing Program for phase contrast microscopy (PCM).

(iii) Until the National Bureau of Standards TEM laboratory accreditation program is operational, local educational agencies shall use laboratories that use the protocol described in appendix A to subpart E of this part.

(3) Except as provided in paragraphs (i)(4), and (i)(5), of this section, an action to remove, encapsulate, or enclose ACBM shall be considered complete when the average concentration of asbestos of five air samples collected within the affected functional space and analyzed by the TEM method in appendix A of this subpart E, is not statistically significantly different, as determined by the Z-test calculation found in appendix A of this subpart E, from the average asbestos concentration of five air samples collected at the same time outside the affected functional space and analyzed in the same manner, and the average asbestos concentration of the three field blanks described in appendix A of this subpart E is below the filter background level, as defined in appendix A of this subpart E, of 70 structures per square millimeter (70 s/mm^2).

(4) An action may also be considered complete if the volume of air drawn for each of the five samples collected within the affected functional space is equal to or greater than 1,199 L of air for a 25 mm filter or equal to or greater than 2,799 L of air for a 37 mm filter, and the average concentration of asbestos as analyzed by the TEM method in appendix A of this subpart E, for the five air samples does not exceed the filter background level, as defined in appendix A, of 70 structures per square millimeter (70 s/mm^2). If the average concentration of asbestos of the five air samples within the affected functional space exceeds 70 s/mm^2, or if the volume of air in each of the samples is less than 1,199 L of air for a 25 mm filter or less than 2,799 L of air for a 37 mm filter, the action shall be considered complete only when the requirements of paragraph (i)(3) or (i)(5), of this section are met.

(5) At any time, a local education agency may analyze air monitoring samples collected for clearance purposes by phase contrast microscopy (PCM) to confirm completion of removal, encapsulation, or enclosure of ACBM that is greater than small-scale, short-duration and less than or equal to 160 square feet or 260 linear feet. The action shall be considered complete when the results of samples collected in the affected functional space and analyzed by phase contrast microscopy using the National Institute for Occupational Safety and Health (NIOSH) Method 7400 entitled "Fibers" published in the NIOSH Manual of Analytical Methods, 3rd Edition, Second Supplement, August 1987, show that the concentration of fibers for each of the five samples is less than or equal to a limit of quantitation for PCM (0.01 fibers per cubic centimeter (0.01 f/cm^3) of air). The method is available for public inspection at the Office of the Federal Register, 800 North Capitol Street, NW, Suite 700, Washington, DC, 20408, and the Non-Confidential Information Center (NCIC) (7407), Office of Pollution Prevention and Toxics, U.S. Environmental Protection Agency, Room B–607 NEM, 401 M Street, SW., Washington, DC, 20460, between the hours of 12 p.m. and 4 p.m. weekdays excluding legal holidays. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. The method is incorporated as it exists on the effective date of this rule, and a notice of any change to the method will be published in the FEDERAL REGISTER.

(6) To determine the amount of ACBM affected under paragraph (i)(5) of this section, the local education agency shall add the total square or linear footage of ACBM within the containment barriers used to isolate the functional space for the action to remove, encapsulate, or enclose the ACBM. Contiguous portions of material subject to such action conducted concurrently or at approximately the same time within the same school building shall not be separated to qualify under paragraph (i)(5), of this section.

[52 FR 41846, Oct. 30, 1987, as amended at 53 FR 12525, Apr. 15, 1988; 60 FR 31922, June 19, 1995; 60 FR 34465, July 3, 1995]

§ 763.91 Operations and maintenance.

(a) *Applicability.* The local education agency shall implement an operations, maintenance, and repair (O&M) program under this section whenever any friable ACBM is present or assumed to be present in a building that it leases, owns, or otherwise uses as a school building. Any material identified as nonfriable ACBM or nonfriable assumed ACBM must be treated as friable ACBM for purposes of this section when the material is about to become friable as a result of activities performed in the school building.

(b) *Worker protection.* The protection provided by EPA at 40 CFR 763.121 for worker protection during asbestos abatement projects is extended to employees of local education agencies who perform operations, maintenance, and repair (O&M) activities involving ACM and who are not covered by the OSHA asbestos construction standard at 29 CFR 1926.58 or an asbestos worker approved by OSHA under section 19 of the Occupational Safety and Health Act. Local education agencies may consult appendix B of this subpart if their employees are performing operations, maintenance, and repair activities that are of small-scale, short-duration.

(c) *Cleaning*—(1) *Initial cleaning.* Unless the building has been cleaned using equivalent methods within the previous 6 months, all areas of a school building where friable ACBM, damaged or significantly damaged thermal system insulation ACM, or friable suspected ACBM assumed to be ACM are present shall be cleaned at least once after the completion of the inspection required by § 763.85(a) and before the initiation of any response action, other than O&M activities or repair, according to the following procedures:

(i) HEPA-vacuum or steam-clean all carpets.

(ii) HEPA-vacuum or wet-clean all other floors and all other horizontal surfaces.

(iii) Dispose of all debris, filters, mopheads, and cloths in sealed, leak-tight containers.

(2) *Additional cleaning.* The accredited management planner shall make a written recommendation to the local education agency whether additional cleaning is needed, and if so, the methods and frequency of such cleaning.

(d) *Operations and maintenance activities.* The local education agency shall ensure that the procedures described below to protect building occupants shall be followed for any operations and maintenance activities disturbing friable ACBM:

(1) Restrict entry into the area by persons other than those necessary to perform the maintenance project, either by physically isolating the area or by scheduling.

(2) Post signs to prevent entry by unauthorized persons.

(3) Shut off or temporarily modify the air-handling system and restrict other sources of air movement.

(4) Use work practices or other controls, such as, wet methods, protective clothing, HEPA-vacuums, mini-enclosures, glove bags, as necessary to inhibit the spread of any released fibers.

(5) Clean all fixtures or other components in the immediate work area.

(6) Place the asbestos debris and other cleaning materials in a sealed, leak-tight container.

(e) *Maintenance activities other than small-scale, short-duration.* The response action for any maintenance activities disturbing friable ACBM, other than small-scale, short-duration maintenance activities, shall be designed by persons accredited to design response actions and conducted by persons accredited to conduct response actions.

(f) *Fiber release episodes*—(1) *Minor fiber release episode.* The local education agency shall ensure that the procedures described below are followed in the event of a minor fiber release episode (i.e., the falling or dislodging of 3 square or linear feet or less of friable ACBM): 5

(i) Thoroughly saturate the debris using wet methods.

(ii) Clean the area, as described in paragraph (e) of this section.

(iii) Place the asbestos debris in a sealed, leak-tight container.

(iv) Repair the area of damaged ACM with materials such as asbestos-free spackling, plaster, cement, or insulation, or seal with latex paint or an encapsulant, or immediately have the appropriate response action implemented as required by § 763.90.

(2) *Major fiber release episode.* The local education agency shall ensure that the procedures described below are followed in the event of a major fiber release episode (i.e., the falling or dislodging of more than 3 square or linear feet of friable ACBM):

(i) Restrict entry into the area and post signs to prevent entry into the area by persons other than those necessary to perform the response action.

(ii) Shut off or temporarily modify the air-handling system to prevent the distribution of fibers to other areas in the building.

(iii) The response action for any major fiber release episode must be designed by persons accredited to design response actions and conducted by persons accredited to conduct response actions.

§ 763.92 Training and periodic surveillance.

(a) *Training.* (1) The local education agency shall ensure, prior to the implementation of the O&M provisions of the management plan, that all members of its maintenance and custodial staff (custodians, electricians, heating/air conditioning engineers, plumbers, etc.) who may work in a building that contains ACBM receive awareness training of at least 2 hours, whether or not they are required to work with ACBM. New custodial and maintenance employees shall be trained within 60 days after commencement of employment. Training shall include, but not be limited to:

(i) Information regarding asbestos and its various uses and forms.

(ii) Information on the health effects associated with asbestos exposure.

(iii) Locations of ACBM identified throughout each school building in which they work.

(iv) Recognition of damage, deterioration, and delamination of ACBM.

(v) Name and telephone number of the person designated to carry out general local education agency responsibilities under § 763.84 and the availability and location of the management plan.

(2) The local education agency shall ensure that all members of its maintenance and custodial staff who conduct any activities that will result in the disturbance of ACBM shall receive training described in paragraph (a)(1) of this section and 14 hours of additional training. Additional training shall include, but not be limited to:

(i) Descriptions of the proper methods of handling ACBM.

(ii) Information on the use of respiratory protection as contained in the EPA/NIOSH *Guide to Respiratory Protection for the Asbestos Abatement Industry*, September 1986 (EPA 560/OPPTS-86-001), available from the Director, Environmental Assistance Division (7408), Office of Pollution Prevention and Toxics, U.S. Environmental Protection Agency, Room E-543B, 401 M St., SW., Washington, DC, 20460, Telephone: (202) 554-1404, TDD: (202) 544-0551 and other personal protection measures.

(iii) The provisions of this section and §763.91, Appendices A, B, C, D of this subpart E of this part, EPA regulations contained in 40 CFR part 763, subpart G, and in 40 CFR part 61, subpart M, and OSHA regulations contained in 29 CFR 1926.58.

(iv) Hands-on training in the use of respiratory protection, other personal protection measures, and good work practices.

(3) Local education agency maintenance and custodial staff who have attended EPA-approved asbestos training or received equivalent training for O&M and periodic surveillance activities involving asbestos shall be considered trained for the purposes of this section.

(b) *Periodic surveillance.* (1) At least once every 6 months after a management plan is in effect, each local education agency shall conduct periodic surveillance in each building that it leases, owns, or otherwise uses as a school building that contains ACBM or is assumed to contain ACBM.

(2) Each person performing periodic surveillance shall:

(i) Visually inspect all areas that are identified in the management plan as ACBM or assumed ACBM.

(ii) Record the date of the surveillance, his or her name, and any changes in the condition of the materials.

(iii) Submit to the person designated to carry out general local education agency responsibilities under § 763.84 a copy of such record for inclusion in the management plan.

[52 FR 41846, Oct. 30, 1987, as amended at 60 FR 34465, July 3, 1995]

§ 763.93 Management plans.

(a)(1) On or before October 12, 1988, each local education agency shall develop an asbestos management plan for each school, including all buildings that they lease, own, or otherwise use as school buildings, and submit the plan to an Agency designated by the Governor of the State in which the local education agency is located. The plan may be submitted in stages that cover a portion of the school buildings under the authority of the local education agency.

(2) If a building to be used as part of a school is leased or otherwise acquired after October 12, 1988, the local education agency shall include the new building in the management plan for the school prior to its use as a school building. The revised portions of the management plan shall be submitted to the Agency designated by the Governor.

(3) If a local education agency begins to use a building as a school after October 12, 1988, the local education agency shall submit a management plan for

the school to the Agency designated by the Governor prior to its use as a school.

(b) On or before October 17, 1987, the Governor of each State shall notify local education agencies in the State regarding where to submit their management plans. States may establish administrative procedures for reviewing management plans. If the Governor does not disapprove a management plan within 90 days after receipt of the plan, the local education agency shall implement the plan.

(c) Each local education agency must begin implementation of its management plan on or before July 9, 1989, and complete implementation in a timely fashion.

(d) Each local education agency shall maintain and update its management plan to keep it current with ongoing operations and maintenance, periodic surveillance, inspection, reinspection, and response action activities. All provisions required to be included in the management plan under this section shall be retained as part of the management plan, as well as any information that has been revised to bring the plan up-to-date.

(e) The management plan shall be developed by an accredited management planner and shall include:

(1) A list of the name and address of each school building and whether the school building contains friable ACBM, nonfriable ACBM, and friable and nonfriable suspected ACBM assumed to be ACM.

(2) For each inspection conducted before the December 14, 1987:

(i) The date of the inspection.

(ii) A blueprint, diagram, or written description of each school building that identifies clearly each location and approximate square or linear footage of any homogeneous or sampling area where material was sampled for ACM, and, if possible, the exact locations where bulk samples were collected, and the dates of collection.

(iii) A copy of the analyses of any bulk samples, dates of analyses, and a copy of any other laboratory reports pertaining to the analyses.

(iv) A description of any response actions or preventive measures taken to reduce asbestos exposure, including if possible, the names and addresses of all contractors involved, start and completion dates of the work, and results of any air samples analyzed during and upon completion of the work.

(v) A description of assessments, required to be made under § 763.88, of material that was identified before December 14, 1987, as friable ACBM or friable suspected ACBM assumed to be ACM, and the name and signature, State of accreditation, and if applicable, accreditation number of each accredited person making the assessments.

(3) For each inspection and reinspection conducted under § 763.85:

(i) The date of the inspection or reinspection and the name and signature, State of accreditation and, if applicable, the accreditation number of each accredited inspector performing the inspection or reinspection.

(ii) A blueprint, diagram, or written description of each school building that identifies clearly each location and approximate square or linear footage of homogeneous areas where material was sampled for ACM, the exact location where each bulk sample was collected, date of collection, homogeneous areas where friable suspected ACBM is assumed to be ACM, and where nonfriable suspected ACBM is assumed to be ACM.

(iii) A description of the manner used to determine sampling locations, and the name and signature of each accredited inspector collecting samples, the State of accreditation, and if applicable, his or her accreditation number.

(iv) A copy of the analyses of any bulk samples collected and analyzed, the name and address of any laboratory that analyzed bulk samples, a statement that the laboratory meets the applicable requirements of § 763.87(a) the date of analysis, and the name and signature of the person performing the analysis.

(v) A description of assessments, required to be made under § 763.88, of all ACBM and suspected ACBM assumed to be ACM, and the name, signature, State of accreditation, and if applicable, accreditation number of each accredited person making the assessments.

(4) The name, address, and telephone number of the person designated under

§ 763.84 to ensure that the duties of the local education agency are carried out, and the course name, and dates and hours of training taken by that person to carry out the duties.

(5) The recommendations made to the local education agency regarding response actions, under § 763.88(d), the name, signature, State of accreditation of each person making the recommendations, and if applicable, his or her accreditation number.

(6) A detailed description of preventive measures and response actions to be taken, including methods to be used, for any friable ACBM, the locations where such measures and action will be taken, reasons for selecting the response action or preventive measure, and a schedule for beginning and completing each preventive measure and response action.

(7) With respect to the person or persons who inspected for ACBM and who will design or carry out response actions, except for operations and maintenance, with respect to the ACBM, one of the following statements:

(i) If the State has adopted a contractor accreditation program under section 206(b) of Title II of the Act, a statement that the person(s) is accredited under such plan.

(ii) A statement that the local education agency used (or will use) persons who have been accredited by another State which has adopted a contractor accreditation plan under section 206(b) of Title II of the Act or is accredited by an EPA-approved course under section 206(c) of Title II of the Act.

(8) A detailed description in the form of a blueprint, diagram, or in writing of any ACBM or suspected ACBM assumed to be ACM which remains in the school once response actions are undertaken pursuant to § 763.90. This description shall be updated as response actions are completed.

(9) A plan for reinspection under § 763.85, a plan for operations and maintenance activities under § 763.91, and a plan for periodic surveillance under § 763.92, a description of the recommendation made by the management planner regarding additional cleaning under § 763.91(c)(2) as part of an operations and maintenance program, and the response of the local education agency to that recommendation.

(10) A description of steps taken to inform workers and building occupants, or their legal guardians, about inspections, reinspections, response actions, and post-response action activities, including periodic reinspection and surveillance activities that are planned or in progress.

(11) An evaluation of the resources needed to complete response actions successfully and carry out reinspection, operations and maintenance activities, periodic surveillance and training.

(12) With respect to each consultant who contributed to the management plan, the name of the consultant and one of the following statements:

(i) If the State has adopted a contractor accreditation plan under section 206(b) of Title II of the Act, a statement that the consultant is accredited under such plan.

(ii) A statement that the contractor is accredited by another State which has adopted a contractor accreditation plan under section 206(b) of Title II of the Act, or is accredited by an EPA-approved course developed under section 206(c) of Title II of the Act.

(f) A local education agency may require each management plan to contain a statement signed by an accredited management plan developer that such person has prepared or assisted in the preparation of such plan or has reviewed such plan, and that such plan is in compliance with this subpart E. Such statement may not be signed by a person who, in addition to preparing or assisting in preparing the management plan, also implements (or will implement) the management plan.

(g)(1) Upon submission of a management plan to the Governor for review, a local education agency shall keep a copy of the plan in its administrative office. The management plans shall be available, without cost or restriction, for inspection by representatives of EPA and the State, the public, including teachers, other school personnel and their representatives, and parents. The local education agency may charge a reasonable cost to make copies of management plans.

(2) Each local education agency shall maintain in its administrative office a complete, updated copy of a management plan for each school under its administrative control or direction. The management plans shall be available, during normal business hours, without cost or restriction, for inspection by representatives of EPA and the State, the public, including teachers, other school personnel and their representatives, and parents. The local education agency may charge a reasonable cost to make copies of management plans.

(3) Each school shall maintain in its administrative office a complete, updated copy of the management plan for that school. Management plans shall be available for inspection, without cost or restriction, to workers before work begins in any area of a school building. The school shall make management plans available for inspection to representatives of EPA and the State, the public, including parents, teachers, and other school personnel and their representatives within 5 working days after receiving a request for inspection. The school may charge a reasonable cost to make copies of the management plan.

(4) Upon submission of its management plan to the Governor and at least once each school year, the local education agency shall notify in writing parent, teacher, and employee organizations of the availability of management plans and shall include in the management plan a description of the steps taken to notify such organizations, and a dated copy of the notification. In the absence of any such organizations for parents, teachers, or employees, the local education agency shall provide written notice to that relevant group of the availability of management plans and shall include in the management plan a description of the steps taken to notify such groups, and a dated copy of the notification.

(h) Records required under §763.94 shall be made by local education agencies and maintained as part of the management plan.

(i) Each management plan must contain a true and correct statement, signed by the individual designated by the local education agency under §763.84, which certifies that the general, local education agency responsibilities, as stipulated by §763.84, have been met or will be met.

§ 763.94 Recordkeeping.

(a) Records required under this section shall be maintained in a centralized location in the administrative office of both the school and the local education agency as part of the management plan. For each homogeneous area where all ACBM has been removed, the local education agency shall ensure that such records are retained for 3 years after the next reinspection required under §763.85(b)(1), or for an equivalent period.

(b) For each preventive measure and response action taken for friable and nonfriable ACBM and friable and nonfriable suspected ACBM assumed to be ACM, the local education agency shall provide:

(1) A detailed written description of the measure or action, including methods used, the location where the measure or action was taken, reasons for selecting the measure or action, start and completion dates of the work, names and addresses of all contractors involved, and if applicable, their State of accreditation, and accreditation numbers, and if ACBM is removed, the name and location of storage or disposal site of the ACM.

(2) The name and signature of any person collecting any air sample required to be collected at the completion of certain response actions specified by §763.90(i), the locations where samples were collected, date of collection, the name and address of the laboratory analyzing the samples, the date of analysis, the results of the analysis, the method of analysis, the name and signature of the person performing the analysis, and a statement that the laboratory meets the applicable requirements of §763.90(i)(2)(ii).

(c) For each person required to be trained under §763.92(a) (1) and (2), the local education agency shall provide the person's name and job title, the date that training was completed by that person, the location of the training, and the number of hours completed in such training.

(d) For each time that periodic surveillance under §763.92(b) is performed, the local education agency shall record the name of each person performing the surveillance, the date of the surveillance, and any changes in the conditions of the materials.

(e) For each time that cleaning under § 763.91(c) is performed, the local education agency shall record the name of each person performing the cleaning, the date of such cleaning, the locations cleaned, and the methods used to perform such cleaning.

(f) For each time that operations and maintenance activities under § 763.91(d) are performed, the local education agency shall record the name of each person performing the activity, the start and completion dates of the activity, the locations where such activity occurred, a description of the activity including preventive measures used, and if ACBM is removed, the name and location of storage or disposal site of the ACM.

(g) For each time that major asbestos activity under § 763.91(e) is performed, the local education agency shall provide the name and signature, State of accreditation, and if applicable, the accreditation number of each person performing the activity, the start and completion dates of the activity, the locations where such activity occurred, a description of the activity including preventive measures used, and if ACBM is removed, the name and location of storage or disposal site of the ACM.

(h) For each fiber release episode under § 763.91(f), the local education agency shall provide the date and location of the episode, the method of repair, preventive measures or response action taken, the name of each person performing the work, and if ACBM is removed, the name and location of storage or disposal site of the ACM.

(Approved by the Office of Management and Budget under control number 2070–0091)

§ 763.95 Warning labels.

(a) The local education agency shall attach a warning label immediately adjacent to any friable and nonfriable ACBM and suspected ACBM assumed to be ACM located in routine maintenance areas (such as boiler rooms) at each school building. This shall include:

(1) Friable ACBM that was responded to by a means other than removal.

(2) ACBM for which no response action was carried out.

(b) All labels shall be prominently displayed in readily visible locations and shall remain posted until the ACBM that is labeled is removed.

(c) The warning label shall read, in print which is readily visible because of large size or bright color, as follows:

CAUTION: ASBESTOS. HAZARDOUS. DO NOT DISTURB WITHOUT PROPER TRAINING AND EQUIPMENT.

§ 763.97 Compliance and enforcement.

(a) *Compliance with Title II of the Act.* (1) Section 207(a) of Title II of the Act (15 U.S.C. 2647) makes it unlawful for any local education agency to:

(i) Fail to conduct inspections pursuant to section 203(b) of Title II of the Act, including failure to follow procedures and failure to use accredited personnel and laboratories.

(ii) Knowingly submit false information to the Governor regarding any inspection pursuant to regulations under section 203(i) of Title II of the Act.

(iii) Fail to develop a management plan pursuant to regulations under section 203(i) of Title II of the Act.

(2) Section 207(a) of Title II of the Act (15 U.S.C. 2647) also provides that any local education agency which violates any provision of section 207 shall be liable for a civil penalty of not more than $5,000 for each day during which the violation continues. For the purposes of this subpart, a "violation" means a failure to comply with respect to a single school building.

(b) *Compliance with Title I of the Act.* (1) Section 15(1)(D) of Title I of the Act (15 U.S.C. 2614) makes it unlawful for any person to fail or refuse to comply with any requirement of Title II or any rule promulgated or order issued under Title II. Therefore, any person who violates any requirement of this subpart is in violation of section 15 of Title I of the Act.

(2) Section 15(3) of Title I of the Act (15 U.S.C. 2614) makes it unlawful for any person to fail or refuse to establish or maintain records, submit reports, notices or other information, or permit

access to or copying of records, as required by this Act or a rule thereunder.

(3) Section 15(4) (15 U.S.C. 2614) of Title I of the Act makes it unlawful for any person to fail or refuse to permit entry or inspection as required by section 11 of Title I of the Act.

(4) Section 16(a) of Title I of the Act (15 U.S.C. 2615) provides that any person who violates any provision of section 15 of Title I of the Act shall be liable to the United States for a civil penalty in an amount not to exceed $25,000 for each such violation. Each day such a violation continues shall, for purposes of this paragraph, constitute a separate violation of section 15. A local education agency is not liable for any civil penalty under Title I of the Act for failing or refusing to comply with any rule promulgated or order issued under Title II of the Act.

(c) *Criminal penalties.* If any violation committed by any person (including a local education agency) is knowing or willful, criminal penalties may be assessed under section 16(b) of Title I of the Act.

(d) *Injunctive relief.* The Agency may obtain injunctive relief under section 208(b) of Title II of the Act to respond to a hazard which poses an imminent and substantial endangerment to human health or the environment or section 17 (15 U.S.C. 2616) of Title I of the Act to restrain any violation of section 15 of Title I of the Act or to compel the taking of any action required by or under Title I of the Act.

(e) *Citizen complaints.* Any citizen who wishes to file a complaint pursuant to section 207(d) of Title II of the Act should direct the complaint to the Governor of the State or the EPA Asbestos Ombudsman, 401 M Street, SW., Washington, DC 20460. The citizen complaint should be in writing and identified as a citizen complaint pursuant to section 207(d) of Title II of TSCA. The EPA Asbestos Ombudsman or the Governor shall investigate and respond to the complaint within a reasonable period of time if the allegations provide a reasonable basis to believe that a violation of the Act has occurred.

(f) *Inspections.* EPA may conduct inspections and review management plans under section 11 of Title I of the Act (15 U.S.C. 2610) to ensure compliance.

§ 763.98 Waiver; delegation to State.

(a) *General.* (1) Upon request from a State Governor and after notice and comment and an opportunity for a public hearing in accordance with paragraphs (b) and (c) of this section, EPA may waive some or all of the requirements of this subpart E if the State has established and is implementing or intends to implement a program of asbestos inspection and management that contains requirements that are at least as stringent as the requirements of this subpart E.

(2) A waiver from any requirement of this subpart E shall apply only to the specific provision for which a waiver has been granted under this section. All requirements of this subpart E shall apply until a waiver is granted under this section.

(b) *Request.* Each request by a Governor to waive any requirement of this subpart E shall be sent with three complete copies of the request to the Regional Administrator for the EPA Region in which the State is located and shall include:

(1) A copy of the State provisions or proposed provisions relating to its program of asbestos inspection and management in schools for which the request is made.

(2)(i) The name of the State agency that is or will be responsible for administering and enforcing the requirements for which a waiver is requested, the names and job titles of responsible officials in that agency, and phone numbers where the officials can be contacted.

(ii) In the event that more than one agency is or will be responsible for administering and enforcing the requirements for which a waiver is requested, a description of the functions to be performed by each agency, how the program will be coordinated by the lead agency to ensure consistency and effective administration in the asbestos inspection and management program within the State, the names and job titles of responsible officials in the agencies, and phone numbers where the officials can be contacted. The lead agency will serve as the central contact point for the EPA.

(3) Detailed reasons, supporting papers, and the rationale for concluding that the State's asbestos inspection and management program provisions for which the request is made are at least as stringent as the requirements of this subpart E.

(4) A discussion of any special situations, problems, and needs pertaining to the waiver request accompanied by an explanation of how the State intends to handle them.

(5) A statement of the resources that the State intends to devote to the administration and enforcement of the provisions relating to the waiver request.

(6) Copies of any specific or enabling State laws (enacted and pending enactment) and regulations (promulgated and pending promulgation) relating to the request, including provisions for assessing criminal and/or civil penalties.

(7) Assurance from the Governor, the Attorney General, or the legal counsel of the lead agency that the lead agency or other cooperating agencies have the legal authority necessary to carry out the requirements relating to the request.

(c) *General notice—hearing.* (1) Within 30 days after receipt of a request for a waiver, EPA will determine the completeness of the request. If EPA does not request further information within the 30-day period, the request will be deemed complete.

(2) Within 30 days after EPA determines that a request is complete, EPA will issue for publication in the FEDERAL REGISTER a notice that announces receipt of the request, describes the information submitted under paragraph (b) of this section, and solicits written comment from interested members of the public. Comments must be submitted within 60 days.

(3) If, during the comment period, EPA receives a written objection to a Governor's request and a request for a public hearing detailing specific objections to the granting of a waiver, EPA will schedule a public hearing to be held in the affected State after the close of the comment period and will announce the public hearing date in the FEDERAL REGISTER before the date of the hearing. Each comment shall include the name and address of the person submitting the comment.

(d) *Criteria.* EPA may waive some or all of the requirements of subpart E of this part if:

(1) The State's lead agency and other cooperating agencies have the legal authority necessary to carry out the provisions of asbestos inspection and management in schools relating to the waiver request.

(2) The State's program of asbestos inspection and management in schools relating to the waiver request and implementation of the program are or will be at least as stringent as the requirements of this subpart E.

(3) The State has an enforcement mechanism to allow it to implement the program described in the waiver request.

(4) The lead agency and any cooperating agencies have or will have qualified personnel to carry out the provisions relating to the waiver request.

(5) The State will devote adequate resources to the administration and enforcement of the asbestos inspection and management provisions relating to the waiver request.

(6) When specified by EPA, the State gives satisfactory assurances that necessary steps, including specific actions it proposes to take and a time schedule for their accomplishment, will be taken within a reasonable time to conform with applicable criteria under paragraphs (d) (2) through (4) of this section.

(e) *Decision.* EPA will issue for publication in the FEDERAL REGISTER a notice announcing its decision to grant or deny, in whole or in part, a Governor's request for a waiver from some or all of the requirements of this subpart E within 30 days after the close of the comment period or within 30 days following a public hearing, whichever is applicable. The notice will include the Agency's reasons and rationale for granting or denying the Governor's request. The 30-day period may be extended if mutually agreed upon by EPA and the State.

(f) *Modifications.* When any substantial change is made in the administration or enforcement of a State program for which a waiver was granted under this section, a responsible official in the lead agency shall submit such changes to EPA.

(g) *Reports.* The lead agency in each State that has been granted a waiver by EPA from any requirement of subpart E of this part shall submit a report to the Regional Administrator for the Region in which the State is located at least once every 12 months to include the following information:

(1) A summary of the State's implementation and enforcement activities during the last reporting period relating to provisions waived under this section, including enforcement actions taken.

(2) Any changes in the administration or enforcement of the State program implemented during the last reporting period.

(3) Other reports as may be required by EPA to carry out effective oversight of any requirement of this subpart E that was waived under this section.

(h) *Oversight.* EPA may periodically evaluate the adequacy of a State's implementation and enforcement of and resources devoted to carrying out requirements relating to the waiver. This evaluation may include, but is not limited to, site visits to local education agencies without prior notice to the State.

(i) *Informal conference.* (1) EPA may request that an informal conference be held between appropriate State and EPA officials when EPA has reason to believe that a State has failed to:

(i) Substantially comply with the terms of any provision that was waived under this section.

(ii) Meet the criteria under paragraph (d) of this section, including the failure to carry out enforcement activities or act on violations of the State program.

(2) EPA will:

(i) Specify to the State those aspects of the State's program believed to be inadequate.

(ii) Specify to the State the facts that underlie the belief of inadequacy.

(3) If EPA finds, on the basis of information submitted by the State at the conference, that deficiencies did not exist or were corrected by the State, no further action is required.

(4) Where EPA finds that deficiencies in the State program exist, a plan to correct the deficiencies shall be negotiated between the State and EPA. The plan shall detail the deficiencies found in the State program, specify the steps the State has taken or will take to remedy the deficiencies, and establish a schedule for each remedial action to be initiated.

(j) *Rescission.* (1) If the State fails to meet with EPA or fails to correct deficiencies raised at the informal conference, EPA will deliver to the Governor of the State and a responsible official in the lead agency a written notice of its intent to rescind, in whole or part, the waiver.

(2) EPA will issue for publication in the FEDERAL REGISTER a notice that announces the rescission of the waiver, describes those aspects of the State's program determined to be inadequate, and specifies the facts that underlie the findings of inadequacy.

§ 763.99 Exclusions.

(a) A local education agency shall not be required to perform an inspection under § 763.85(a) in any sampling area as defined in 40 CFR 763.103 or homogeneous area of a school building where:

(1) An accredited inspector has determined that, based on sampling records, friable ACBM was identified in that homogeneous or sampling area during an inspection conducted before December 14, 1987. The inspector shall sign and date a statement to that effect with his or her State of accreditation and if applicable, accreditation number and, within 30 days after such determination, submit a copy of the statement to the person designated under § 763.84 for inclusion in the management plan. However, an accredited inspector shall assess the friable ACBM under § 763.88.

(2) An accredited inspector has determined that, based on sampling records, nonfriable ACBM was identified in that homogeneous or sampling area during an inspection conducted before December 14, 1987. The inspector shall sign and date a statement to that effect with his or her State of accreditation and if applicable, accreditation number

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[47 FR 23369, May 27, 1982; 47 FR 38535, Sept. 1, 1982; Redesignated at 60 FR 31922, June 19, 1995]

Subpart F [Reserved]

Subpart G—Asbestos Abatement Projects

SOURCE: 52 FR 5623, Feb. 25, 1987, unless otherwise noted.

§ 763.120 Scope

(a) This part establishes requirements which must be followed during asbestos abatement projects by employers of State and local government employees not covered by the Asbestos Standard of the Occupational Safety and Health Administration (OSHA), 29 CFR 1926.58, an Asbestos Standard adopted by a State as part of a State plan approved by OSHA under section 18 of the Occupational Safety and Health Act, or a State asbestos regulation which EPA has determined to be comparable to or more stringent than this part. The rule covers those employees who take part in asbestos abatement work.

(b) [Reserved]

§ 763.121 Regulatory requirements.

(a) [Reserved]

(b) *Definitions. Action level* means an airborne concentration of asbestos of 0.1 fiber per cubic centimeter (f/cc) of air calculated as an 8-hour time-weighted average.

Administrator means the Administrator, U.S. Environmental Protection Agency, or designee.

Asbestos means the asbestiform varieties of chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite—grunerite); tremolite; anthophyllite, and actinolite.

Asbestos abatement project means any activity involving the removal, enclosure, or encapsulation of friable asbestos material.

Authorized person means any person authorized by the employer and required by work duties to be present in regulated areas.

Clean room means an uncontaminated room having facilities for the storage of employees' street clothing and uncontaminated materials and equipment.

Competent person means one who is capable of identifying existing asbestos hazards in the workplace and who has the authority to take prompt corrective measures to eliminate them. The duties of the competent person include at least the following: Establishing the negative-pressure enclosure, ensuring its integrity, and controlling entry to and exit from the enclosure; supervising any employee exposure monitoring required by this subpart, ensuring that all employees working within such an enclosure wear the appropriate personal protective equipment, are trained in the use of appropriate methods of exposure control, and use the hygiene facilities and decontamination procedures specified in this subpart; and ensuring that engineering controls in use are in proper operating condition and are functioning properly.

Decontamination area means an enclosed area adjacent and connected to the regulated area and consisting of an equipment room, shower area, and clean room, which is used for the decontamination of workers, materials, and equipment contaminated with asbestos.

	Yes or No	Further Comment on Positive Answers
Pneumonia	______	
Tuberculosis	______	
Chest Surgery	______	
Other Lung Problems	______	
Heart Disease	______	
Do you have:		
Frequent colds	______	
Chronic cough	______	
Shortness of breath when walking or climbing one flight of stairs	______	
Do you:		
Wheeze	______	
Cough up phlegm	______	
Smoke cigarettes	______	Packs per day ___ How many years ___

Date ________________ Signature ________________________

APPENDIX E TO § 763.121—INTERPRETATION AND CLASSIFICATION OF CHEST ROENTGENOGRAMS—MANDATORY

(a) Chest roentgenograms shall be interpreted and classified in accordance with a professionally accepted classification system and recorded on an interpretation form following the format of the CDC/NIOSH (M) 2.8 form. As a minimum, the content within the boldlines of this form (items 1 through 4) shall be included. This form is not to be submitted to NIOSH.

(b) Roentgenograms shall be interpreted and classified only by a B-reader, a board eligible/certified radiologist, or an experienced physician with known expertise in pneumoconioses.

(c) All interpreters, whenever interpreting chest roentgenograms made under this section, shall have immediately available for reference a complete set of the ILO-U/C *International Classification of Radiographs for Pneumoconioses*, 1980.

[52 FR 5623, Feb. 25, 1987; 52 FR 10817, Mar. 30, 1987; 53 FR 1022, Jan. 15, 1988]

§ 763.122 Exclusions for States.

(a) The States of Idaho, Kansas, Oklahoma, and Wisconsin have 6 months or such other reasonable time as suggested by the particular State and approved by the Director of the Office of Pollution Prevention and Toxics to make their regulations comparable to or more stringent than this part, and to submit their regulations to EPA's Office of Pollution Prevention and Toxics for review. If in such reasonable time after March 27, 1987, any of these States have not so revised their regulations and submitted them to EPA, State and local government employees in such States shall be covered by the requirements of this part.

(b) Any other State that wishes to be excluded from this rule shall send a copy of a regulation which it considers to be comparable to or more stringent than this part to EPA's Office of Pollution Prevention and Toxics for review. EPA will review the regulation and tentatively determine whether the regulation is comparable to or more stringent than this part. If EPA makes a positive tentative determination, EPA will propose an amendment to this rule excluding that State from coverage. Interested persons may comment on the proposed exclusion during the period for public comment. After considering any comments, EPA may promulgate the final amendment to the rule.

§ 763.124 Reporting.

(a) Employers subject to this rule must report to the Regional Asbestos Coordinator for the EPA Region in which the asbestos abatement project is located at least 10 days before they begin any asbestos abatement project, except one that involves less than either 3 linear feet or 3 square feet of friable asbestos material, and an emergency project. Employers must report any emergency project covered by this rule as soon as possible but in no case more than 48 hours after the project begins. A list of the EPA Regional Offices is given under § 1.7(b) of this chapter.

(b) The report must include:

(1) The employer's name and address.

(2) The location, including street address, of the asbestos abatement project.

(3) The scheduled starting and completion dates for the asbestos abatement project.

(c) If a report is mailed to EPA, the report must be postmarked at least 10 days before the asbestos abatement project begins unless the report is for an emergency project. In such a case, the report must be postmarked as soon as possible but in no case more than 48 hours after the project begins.

(d) Employers do not have to report under this section if they submit a notice to EPA under the National Emission Standard for Asbestos, § 61.146 of this chapter, at least 10 days before they begin the asbestos abatement project and that notice clearly indicates that employees covered by this rule will perform some or all of the asbestos abatement work.

[47 FR 23369, May 27, 1982, as amended at 58 FR 34205, June 23, 1993]

§ 763.125 Enforcement.

(a) Failure to comply with any provision of this part is a violation of section 15 of the Act (15 U.S.C. 2614).

(b) Failure or refusal to establish and maintain records or to permit access to or copying of records, as required by the Act, is a violation of section 15 of the Act (15 U.S.C. 2614).

(c) Failure or refusal to permit entry or inspection as required by section 11 of the Act (15 U.S.C. 2610) is a violation of section 15 of the Act (15 U.S.C. 2614).

(d) Violators may be subject to the civil and criminal penalties in section 16 of the Act (15 U.S.C. 2615) for each violation.

(e) EPA may seek to enjoin an asbestos abatement project in violation of this part, or take other actions under the authority of section 7 or 17 of the Act (15 U.S.C. 2606 or 2616).

§ 763.126 Inspections.

EPA will conduct inspections under section 11 of the Act (15 U.S.C. 2610) to ensure compliance with this part.

Subpart H [Reserved]

Subpart I—Prohibition of the Manufacture, Importation, Processing, and Distribution in Commerce of Certain Asbestos-Containing Products; Labeling Requirements

SOURCE: 54 FR 29507, July 12, 1989, unless otherwise noted.

§ 763.160 Scope.

This subpart prohibits the manufacture, importation, processing, and distribution in commerce of the asbestos-containing products identified and at the dates indicated in §§ 763.165, 763.167, and 763.169. This subpart requires that products subject to this rule's bans, but not yet subject to a ban on distribution in commerce, be labeled. This subpart also includes general exemptions and procedures for requesting exemptions from the provisions of this subpart.

§ 763.163 Definitions.

For purposes of this subpart:

Act means the Toxic Substances Control Act, 15 U.S.C. 2601 *et seq.*

Agency means the United States Environmental Protection Agency.

Asbestos means the asbestiform varieties of: chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite-grunerite); tremolite; anthophyllite; and actinolite.

Asbestos-containing product means any product to which asbestos is deliberately added in any concentration or which contains more than 1.0 percent asbestos by weight or area.

Chemical substance, has the same meaning as in section 3 of the Act.

Commerce has the same meaning as in section 3 of the Act.

Commercial paper means an asbestos-containing product which is made of paper intended for use as general insulation paper or muffler paper. Major applications of commercial papers are insulation against fire, heat transfer, and corrosion in circumstances that require a thin, but durable, barrier.

Corrugated paper means an asbestos-containing product made of corrugated paper, which is often cemented to a flat backing, may be laminated with foils or other materials, and has a corrugated surface. Major applications of asbestos corrugated paper include: thermal insulation for pipe coverings; block insulation; panel insulation in elevators; insulation in appliances; and insulation in low-pressure steam, hot water, and process lines.

Customs territory of the United States means the 50 States, Puerto Rico, and the District of Columbia.

Distribute in commerce has the same meaning as in section 3 of the Act, but the term does not include actions taken with respect to an asbestos-containing product (to sell, resale, deliver, or hold) in connection with the end use of the product by persons who are users (persons who use the product for its intended purpose after it is manufactured or processed). The term also does not include distribution by manufacturers, importers, and processors, and other persons solely for purposes of disposal of an asbestos-containing product.

Flooring felt means an asbestos-containing product which is made of paper felt intended for use as an underlayer for floor coverings, or to be bonded to the underside of vinyl sheet flooring.

Import means to bring into the customs territory of the United States, except for: (1) Shipment through the customs territory of the United States for export without any use, processing, or disposal within the customs territory of the United States; or (2) entering the customs territory of the United States as a component of a product during normal personal or business activities involving use of the product.

Importer means anyone who imports a chemical substance, including a chemical substance as part of a mixture or article, into the customs territory of the United States. *Importer* includes the person primarily liable for the payment of any duties on the merchandise or an authorized agent acting on his or her behalf. The term includes as appropriate:

(1) The consignee.

(2) The importer of record.

(3) The actual owner if an actual owner's declaration and superseding bond has been filed in accordance with 19 CFR 141.20.

(4) The transferee, if the right to withdraw merchandise in a bonded warehouse has been transferred in accordance with subpart C of 19 CFR part 144.

Manufacture means to produce or manufacture in the United States.

Manufacturer means a person who produces or manufactures in the United States.

New uses of asbestos means commercial uses of asbestos not identified in §763.165 the manufacture, importation or processing of which would be initiated for the first time after August 25, 1989.

Person means any natural person, firm, company, corporation, joint-venture, partnership, sole proprietorship, association, or any other business entity; any State or political subdivision thereof, or any municipality; any interstate body and any department, agency, or instrumentality of the Federal Government.

Process has the same meaning as in section 3 of the Act.

Processor has the same meaning as in section 3 of the Act.

Rollboard means an asbestos-containing product made of paper that is produced in a continuous sheet, is flexible, and is rolled to achieve a desired thickness. Asbestos rollboard consists of two sheets of asbestos paper laminated together. Major applications of this product include: office partitioning; garage paneling; linings for stoves and electric switch boxes; and fire-proofing agent for security boxes, safes, and files.

Specialty paper means an asbestos-containing product that is made of paper intended for use as filters for beverages or other fluids or as paper fill for cooling towers. Cooling tower fill consists of asbestos paper that is used as a cooling agent for liquids from industrial processes and air conditioning systems.

State has the same meaning as in section 3 of the Act.

Stock-on-hand means the products which are in the possession, direction, or control of a person and are intended for distribution in commerce.

United States has the same meaning as in section 3 of the Act.

[59 FR 33208, June 28, 1994]

§ 763.165 Manufacture and importation prohibitions.

(a) After August 27, 1990, no person shall manufacture or import the following asbestos-containing products, either for use in the United States or for export: flooring felt and new uses of asbestos.

(b) After August 26, 1996, no person shall manufacture or import the following asbestos-containing products, either for use in the United States or for export: commercial paper, corrugated paper, rollboard, and specialty paper.

(c) The import prohibitions of this subpart do not prohibit:

(1) The import into the customs territory of the United States of products imported solely for shipment outside the customs territory of the United States, unless further repackaging or processing of the product is performed in the United States; or

(2) Activities involving purchases or acquisitions of small quantities of products made outside the customs territory of the United States for personal use in the United States.

[59 FR 33209, June 28, 1994]

§ 763.167 Processing prohibitions.

(a) After August 27, 1990, no person shall process for any use, either in the United States or for export, any of the asbestos-containing products listed at § 763.165(a).

(b) After August 26, 1996, no person shall process for any use, either in the United States or for export, any of the asbestos-containing products listed at § 763.165(b).

[59 FR 33209, June 28, 1994]

§ 763.169 Distribution in commerce prohibitions.

(a) After August 25, 1992, no person shall distribute in commerce, either for use in the United States or for export, any of the asbestos-containing products listed at § 763.165(a).

(b) After August 25, 1997, no person shall distribute in commerce, either for use in the United States or for export, any of the asbestos-containing products listed at § 763.165(b).

(c) A manufacturer, importer, processor, or any other person who is subject to a ban on distribution in commerce in paragraph (a) or (b) of this section must, within 6 months of the effective date of the ban of a specific asbestos-containing product from distribution in commerce, dispose of all their remaining stock-on-hand of that product, by means that are in compliance with applicable local, State, and Federal restrictions which are current at that time.

[59 FR 33209, June 28, 1994]

§ 763.171 Labeling requirements.

(a) After August 27, 1990, manufacturers, importers, and processors of all asbestos-containing products that are identified in § 763.165(a) shall label the products as specified in this subpart at the time of manufacture, import, or processing. This requirement includes labeling all manufacturers', importers', and processors' stock-on-hand as of August 27, 1990.

(b) After August 25, 1995, manufacturers, importers, and processors of all asbestos-containing products that are identified in § 763.165(b), shall label the products as specified in this subpart at the time of manufacture, import, or processing. This requirement includes labeling all manufacturers', importers', and processors' stock-on-hand as of August 25, 1995.

(c) The label shall be placed directly on the visible exterior of the wrappings and packaging in which the product is placed for sale, shipment, or storage. If the product has more than one layer of external wrapping or packaging, the label must be attached to the innermost layer adjacent to the product. If the innermost layer of product wrapping or packaging does not have a visible exterior surface larger than 5 square inches, either a tag meeting the requirements of paragraph (d) of this section must be securely attached to the product's innermost layer of product wrapping or packaging, or a label must be attached to the next outer layer of product packaging or wrapping. Any products that are distributed in commerce to someone other than the end user, shipped, or stored without packaging or wrapping must be labeled or tagged directly on a visible exterior surface of the product as described in paragraph (d) of this section.

(d)(1) Labels must be either printed directly on product packaging or in the form of a sticker or tag made of plastic, paper, metal, or other durable substances. Labels must be attached in such a manner that they cannot be removed without defacing or destroying them. Product labels shall appear as in paragraph (d)(2) of this section and consist of block letters and numerals of color that contrasts with the background of the label or tag. Labels shall be sufficiently durable to equal or exceed the life, including storage and disposal, of the product packaging or wrapping. The size of the label or tag must be at least 15.25 cm (6 inches) on each side. If the product packaging is too small to accommodate a label of this size, the label may be reduced in size proportionately to the size of the product packaging or wrapping down to a minimum 2.5 cm (1 inch) on each side if the product wrapping or packaging has a visible exterior surface larger than 5 square inches.

(2) Products subject to this subpart shall be labeled in English as follows:

NOTICE

This product contains *ASBESTOS*. The U.S. Environmental Protection Agency has banned the distribution in U.S. commerce of this product under section 6 of the Toxic Substances Control Act (15 U.S.C. 2605) as of (insert effective date of ban on distribution in commerce). Distribution of this product in commerce after this date and intentionally removing or tampering with this label are *violations of Federal law*.

(e) No one may intentionally remove, deface, cover, or otherwise obscure or tamper with a label or sticker that has been applied in compliance with this section, except when the product is used or disposed of.

[59 FR 33209, June 28, 1994]

§ 763.173 Exemptions.

(a) Persons who are subject to the prohibitions imposed by §§ 763.165, 763.167, or 763.169 may file an application for an exemption. Persons whose exemption applications are approved by the Agency may manufacture, import, process, or distribute in commerce the banned product as specified in the Agency's approval of the application. No applicant for an exemption may continue the banned activity that is the subject of an exemption application after the effective date of the ban unless the Agency has granted the exemption or the applicant receives an extension under paragraph (b)(4) or (5) of this section.

(b) Application filing dates. (1) Applications for products affected by the prohibitions under §§ 763.165(a) and 763.167(a) may be submitted at any time and will be either granted or denied by EPA as soon as is feasible.

(2) Applications for products affected by the ban under § 763.169(a) may be submitted at any time and will be either granted or denied by EPA as soon as is feasible.

(3) Applications for products affected by the ban under §§ 763.165(b) and 763.167(b) may not be submitted prior to February 27, 1995. Complete applications received after that date, but before August 25, 1995, will be either granted or denied by the Agency prior to the effective date of the ban for the

product. Applications received after August 25, 1995, will be either granted or denied by EPA as soon as is feasible.

(4) Applications for products affected by the ban under § 763.169(b) may not be submitted prior to February 26, 1996. Complete applications received after that date, but before August 26, 1996, will be either granted or denied by the Agency prior to the effective date of the ban for the product. Applications received after August 26, 1996, will be either granted or denied by EPA as soon as is feasible.

(5) The Agency will consider an application for an exemption from a ban under § 763.169 for a product at the same time the applicant submits an application for an exemption from a ban under § 763.165 or § 763.167 for that product. EPA will grant an exemption at that time from a ban under § 763.169 if the Agency determines it appropriate to do so.

(6) If the Agency denies an application less than 30 days before the effective date of a ban for a product, the applicant can continue the activity for 30 days after receipt of the denial from the Agency.

(7) If the Agency fails to meet the deadlines stated in paragraphs (b)(3) and (b)(4) of this section for granting or denying a complete application in instances in which the deadline is before the effective date of the ban to which the application applies, the applicant will be granted an extension of 1 year from the Agency's deadline date. During this extension period the applicant may continue the activity that is the subject of the exemption application. The Agency will either grant or deny the application during the extension period. The extension period will terminate either on the date the Agency grants the application or 30 days after the applicant receives the Agency's denial of the application. However, no extension will be granted if the Agency is scheduled to grant or deny an application at some date after the effective date of the ban, pursuant to the deadlines stated in paragraphs (b)(3) and (b)(4) of this section.

(c) *Where to file.* All applications must be submitted to the following location: TSCA Docket Receipts Office (7407), Office of Pollution Prevention and Toxics, U.S. Environmental Protection Agency, Rm E-G99, 401 M St., SW., Washington, DC 20460, ATTENTION: Asbestos Exemption. For information regarding the submission of exemptions containing information claimed as confidential business information (CBI), see § 763.179.

(d) *Content of application and criteria for decisionmaking.*

(1) *Content of application.* Each application must contain the following:

(i) Name, address, and telephone number of the applicant.

(ii) Description of the manufacturing, import, processing, and/or distribution in commerce activity for which an exemption is requested, including a description of the asbestos-containing product to be manufactured, imported, processed, or distributed in commerce.

(iii) Identification of locations at which the exempted activity would take place.

(iv) Length of time requested for exemption (maximum length of an exemption is 4 years).

(v) Estimated amount of asbestos to be used in the activity that is the subject of the exemption application.

(vi) Data demonstrating the exposure level over the life cycle of the product that is the subject of the application.

(vii) Data concerning:

(A) The extent to which non-asbestos substitutes for the product that is the subject of the application fall significantly short in performance under necessary product standards or requirements, including laws or ordinances mandating product safety standards.

(B) The costs of non-asbestos substitutes relative to the costs of the asbestos-containing product and, in the case in which the product is a component of another product, the effect on the cost of the end use product of using the substitute component.

(C) The extent to which the product or use serves a high-valued use.

(viii) Evidence of demonstrable good faith attempts by the applicant to develop and use a non-asbestos substance or product which may be substituted for the asbestos-containing product or the asbestos in the product or use that is the subject to the application.

(ix) Evidence, in addition to that provided in the other information required with the application, showing that the continued manufacture, importation, processing, distribution in commerce, and use, as applicable, of the product will not present an unreasonable risk of injury to human health.

(2) Criteria for decision (existing products). After considering all the information provided by an applicant under paragraphs (d)(1) and (e) of this section, and any other information available to EPA, EPA will grant an exemption from the prohibitions in §§ 763.165, 763.167, or 763.169 for an applicant's asbestos-containing product only if EPA determines both of the following:

(i) The applicant has made good faith attempts to develop and use a non-asbestos substance or product which may be substituted for the asbestos-containing product or the asbestos in the product or use, and those attempts have failed to produce a substitute or a substitute that results in a product that can be economically produced.

(ii) Continued manufacturing, processing, distribution in commerce, and use, as applicable, of the product will not present an unreasonable risk of injury to human health.

(3) Criteria for decision (new products). Requests to develop and use an asbestos substance or product will be treated as a petition pursuant to section 21 of TSCA.

(e) The Agency reserves the right to request further information from an exemption applicant if necessary to complete the Agency's evaluation of an application.

(f) Upon receipt of a complete application, the Agency will issue a notice in the FEDERAL REGISTER announcing its receipt and invite public comments on the merits of the application.

(g) If the application does not include all of the information required in paragraph (d) of this section, the Agency will return it to the applicant as incomplete and any resubmission of the application will be considered a new application for purposes of the availability of any extension period. If the application is substantially inadequate to allow the Agency to make a reasoned judgment on any of the information required in paragraph (d) of this section and the Agency chooses to request additional information from the applicant, the Agency may also determine that an extension period provided for in paragraph (b)(5) of this section is unavailable to the applicant.

(h) When denying an application, the Agency will notify the applicant by registered mail of its decision and rationale. Whenever possible, the Agency will send this letter prior to the appropriate ban. This letter will be considered a final Agency action for purposes of judicial review. A notice announcing the Agency's denial of the application will be published in the FEDERAL REGISTER.

(i) If the Agency proposes to approve an exemption, it will issue a notice in the FEDERAL REGISTER announcing this intent and invite public comments. If, after considering any timely comments received, the Agency approves an exemption, its decision will be published in the FEDERAL REGISTER. This notice will be considered a final Agency action for purposes of judicial review.

(j) The length of an exemption period will be specified by the agency when it approves the exemption. To extend an exemption period beyond the period stipulated by EPA, applicants must submit a new application to the Agency, following the application procedures described in this section. Applications may not be submitted prior to 15 months before the expiration of the exemption period, unless stated otherwise in the notice granting the exemption. Applications received between 15 months and 1 year before the end of the exemption period will be either granted or denied by the Agency before the end of the exemption period. Applications received after the date 1 year prior to the end of the exemption period will be either granted or denied by the Agency as soon as is feasible. Applicants may not continue the activity that is the subject of the renewal application after the date of the end of the exemption period.

[54 FR 29507, July 12, 1989; 54 FR 37531, Sept. 11, 1989, as amended at 54 FR 46898, Nov. 8, 1989; 59 FR 33210, June 28, 1994]

§ 763.175 Enforcement.

(a) Failure to comply with any provision of this subpart is a violation of section 15 of the Act (15 U.S.C. 2614).

(b) Failure or refusal to establish and maintain records, or to permit access to or copying of records as required by section 11 of the Act (15 U.S.C. 2610) is a violation of section 15 of the Act (15 U.S.C. 2614).

(c) Failure or refusal to permit entry or inspection as required by section 11 of the Act (15 U.S.C. 2610) is a violation of section 15 of the Act (15 U.S.C. 2614).

(d) Violators may be subject to the civil and criminal penalties in section 16 of the Act (15 U.S.C. 2615) for each violation.

(e) The Agency may seek to enjoin the manufacture, import, processing, or distribution in commerce of asbestos-containing products in violation of this subpart, or act to seize any asbestos-containing products manufactured, imported, processed, or distributed in commerce in violation of this subpart, or take any other actions under the authority of section 7 or 17 of the Act (15 U.S.C. 2606 or 2616) that are appropriate.

§ 763.176 Inspections.

The Agency will conduct inspections under section 11 of the Act (15 U.S.C. 2610) to ensure compliance with this subpart.

§ 763.178 Recordkeeping.

(a) *Inventory.* (1) Each person who is subject to the prohibitions imposed by §§ 763.165 and 763.167 must perform an inventory of the stock-on-hand of each banned product as of the effective date of the ban for that product for the applicable activity.

(2) The inventory shall be in writing and shall include the type of product, the number of product units currently in the stock-on-hand of the person performing the inventory, and the location of the stock.

(3) Results of the inventory for a banned product must be maintained by the person for 3 years after the effective date of the § 763.165 or § 763.167 ban on the product.

(b) *Records.* (1) Each person whose activities are subject to the bans imposed by §§ 763.165, 763.167, and 763.169 for a product must, between the effective date of the § 763.165 or § 763.167 ban on the product and the § 763.169 ban on the product, keep records of all commercial transactions regarding the product, including the dates of purchases and sales and the quantities purchased or sold. These records must be maintained for 3 years after the effective date of the § 763.169 ban for the product.

(2) Each person who is subject to the requirements of § 763.171 must, for each product required to be labeled, maintain a copy of the label used in compliance with § 763.171. These records must be maintained for 3 years after the effective date of the ban on distribution in commerce for the product for which the § 763.171 requirements apply.

[54 FR 29507, July 12, 1989, as amended by 54 FR 46898, Nov. 8, 1989; 58 FR 34205, June 23, 1993]

§ 763.179 Confidential business information claims.

(a) Applicants for exemptions under § 763.173 may assert a Confidential Business Information (CBI) claim for information in an exemption application or supplement submitted to the Agency under this subpart only if the claim is asserted in accordance with this section, and release of the information would reveal trade secrets or confidential commercial or financial information, as provided in section 14(a) of the Act. Information covered by a CBI claim will be treated in accordance with the procedures set forth in 40 CFR part 2, subpart B. The Agency will place all information not claimed as CBI in the manner described in this section in a public file without further notice to the applicant.

(b) Applicants may assert CBI claims only at the time they submit a completed exemption application and only in the specified manner. If no such claim accompanies the information when it is received by the Agency, the information may be made available to the public without further notice to the applicant. Submitters that claim information as business confidential must do so by writing the word "Confidential" at the top of the page on which the information appears and by underlining, circling, or placing brackets ([ ]) around the information claimed CBI.

(c) Applicants who assert a CBI claim for submitted information must provide the Agency with two copies of their exemption application. The first copy must be complete and contain all information being claimed as CBI. The second copy must contain only information not claimed as CBI. The Agency will place the second copy of the submission in a public file. Failure to furnish a second copy of the submission when information is claimed as CBI in the first copy will be considered a presumptive waiver of the claim of confidentiality. The Agency will notify the applicant by certified mail that a finding of a presumptive waiver of the claim of confidentiality has been made. The applicant has 30 days from the date of receipt of notification to submit the required second copy. Failure to submit the second copy will cause the Agency to place the first copy in a public file.

(d) Applicants must substantiate all claims of CBI at the time the applicant asserts the claim, i.e., when the exemption application or supplement is submitted, by responding to the questions in paragraph (e) of this section. Failure to provide substantiation of a claim at the time the applicant submits the application will result in a waiver of the CBI claim, and the information may be disclosed to the public without further notice to the applicant.

(e) Applicants who assert any CBI claims must substantiate all claims by providing detailed responses to the following:

(1) Is this information subject to a patent or patent application in the United States or elsewhere? If so, why is confidentiality necessary?

(2) For what period do you assert a claim of confidentiality? If the claim is to extend until a certain event or point in time, please indicate that event or time period. Explain why such information should remain confidential until such point.

(3) Has the information that you are claiming as confidential been disclosed to persons outside of your company? Will it be disclosed to such persons in the future? If so, what restrictions, if any, apply to use or further disclosure of the information?

(4) Briefly describe measures taken by your company to guard against undesired disclosure of the information you are claiming as confidential to others.

(5) Does the information claimed as confidential appear or is it referred to in advertising or promotional materials for the product or the resulting end product, safety data sheets or other similar materials for the product or the resulting end product, professional or trade publications, or any other media available to the public or to your competitors? If you answered yes, indicate where the information appears.

(6) If the Agency disclosed the information you are claiming as confidential to the public, how difficult would it be for the competitor to enter the market for your product? Consider in your answer such constraints as capital and marketing cost, specialized technical expertise, or unusual processes.

(7) Has the Agency, another Federal agency, or a Federal court made any confidentiality determination regarding this information? If so, provide copies of such determinations.

(8) How would your company's competitive position be harmed if the Agency disclosed this information? Why should such harm be considered substantial? Describe the causal relationship between the disclosure and harm.

(9) In light of section 14(b) of TSCA, if you have claimed information from a health and safety study as confidential, do you assert that disclosure of this information would disclose a process used in the manufacturing or processing of a product or information unrelated to the effects of asbestos on human health and the environment? If your answer is yes, explain.

PART 766—DIBENZO-PARA-DIOXINS/DIBENZOFURANS

Subpart A—General Provisions

INTERNATIONAL STANDARD

ISO 10312

First edition
1995-05-01

Ambient air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method

Air ambiant — Détermination des fibres d'amiante — Méthode de microscopie électronique à transmission directe

Reference number
ISO 10312:1995(E)

Contents

International Organization for Standardization
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10312 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

Annexes A, B, C, D, E and F form an integral part of this International Standard. Annexes G, H and J are for information only.

Introduction

This International Standard is applicable to the determination of airborne asbestos in a wide range of ambient air situations, including the interior atmospheres of buildings, and for detailed evaluation of any atmosphere in which asbestos structures are likely to be present. Because the best available medical evidence indicates that the numerical fibre concentration and the fibre sizes are the relevant parameters for evaluation of the inhalation hazards, a fibre counting technique is the only logical approach. Most fibres in ambient atmospheres are not asbestos, and therefore there is a requirement for fibres to be identified. Many airborne asbestos fibres in ambient atmospheres have diameters below the resolution limit of the optical microscope. This International Standard is based on transmission electron microscopy, which has adequate resolution to allow detection of small fibres and is currently the only technique capable of unequivocal identification of the majority of individual fibres of asbestos. Asbestos is often found, not as single fibres, but as very complex, aggregated structures which may or may not be also aggregated with other particles. The fibres found suspended in an ambient atmosphere can often be identified unequivocally, if a sufficient measurement effort is expended. However, if each fibre were to be identified in this way, the analysis would become prohibitively expensive. Because of instrumental deficiencies or because of the nature of the particulate, some fibres cannot be positively identified as asbestos, even though the measurements all indicate that they could be asbestos. Subjective factors therefore contribute to this measurement, and consequently a very precise definition of the procedure for identification and enumeration of asbestos fibres is required. The method specified in this International Standard is designed to provide the best description possible of the nature, numerical concentration, and sizes of asbestos-containing particles found in an air sample. This International Standard is necessarily complex, because the instrumental techniques used are complex, and also because a very detailed and logical procedure must be specified to reduce the subjective aspects of the measurement. The method of data recording specified in this International Standard is designed to allow re-evaluation of the structure counting data as new medical evidence becomes available. All of the feasible specimen preparation techniques result in some modification of the airborne particulate. Even the collection of particles from a three-dimensional airborne dispersion onto a two-dimensional filter surface can be considered a modification of the particulate, and some of the particles in most samples are modified by the specimen preparation procedures. However, the procedures specified in this International Standard are designed to minimize the disturbance of the collected particulate material, and the effect of those disturbances which do occur can be evaluated.

This International Standard describes the method of analysis for a single air filter. However, one of the largest potential errors in characterizing asbestos in ambient atmospheres is associated with the variability between filter samples. For this reason, it is necessary to design a replicate sampling scheme in order to determine this International Standard's accuracy and precision.

Ambient air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method

1 Scope

1.1 Substance determined

This International Standard specifies a reference method using transmission electron microscopy for the determination of the concentration of asbestos structures in ambient atmospheres and includes measurement of the lengths, widths and aspect ratios of the asbestos structures. The method allows determination of the type(s) of asbestos fibres present. The method cannot discriminate between individual fibres of the asbestos and non-asbestos analogues of the same amphibole mineral.

1.2 Type of sample

The method is defined for polycarbonate capillary-pore filters or cellulose ester (either mixed esters of cellulose or cellulose nitrate) filters through which a known volume of air has been drawn. The method is suitable for determination of asbestos in both exterior and building atmospheres.

1.3 Measuring range

The range of concentration which can be determined is 50 structures/mm^2 to 7 000 structures/mm^2 on the filter. The air concentrations represented by these values are a function of the volume of air sampled. There is no lower limit to the dimensions of asbestos fibres which can be detected. In practice, microscopists vary in their ability to detect very small asbestos fibres. Therefore, a minimum length of 0,5 µm has been defined as the shortest fibre to be incorporated in the reported results.

1.4 Limit of detection

The limit of detection theoretically can be lowered indefinitely by filtration of progressively larger volumes of air and by extending the examination of the specimens in the electron microscope. In practice, the lowest achievable limit of detection for a particular area of TEM specimen examined is controlled by the total suspended particulate concentration.

For total suspended particulate concentrations of approximately 10 µg/m^3, corresponding to clean, rural atmospheres, and assuming filtration of 4 000 litres of air, an analytical sensitivity of 0,5 structure/l can be obtained, equivalent to a limit of detection of 1,8 structure/l, if an area of 0,195 mm^2 of the TEM specimens is examined. If higher total suspended particulate concentrations are present, the volume of air filtered must be reduced in order to maintain an acceptable particulate loading on the filter, leading to a proportionate increase in the analytical sensitivity.

Where this is the case, lower limits of detection can be achieved by increasing the area of the TEM specimens that is examined. In order to achieve lower limits of detection for fibres and bundles longer than 5 µm, and for PCM equivalent fibres, lower magnifications are specified which permit more rapid examination of larger areas of the TEM specimens when the examination is limited to these dimensions of fibre. The direct analytical method cannot be used if the general particulate loading of the sample collection filter exceeds approximately 10 µg/cm^2 of filter surface, which corresponds to approximately 10 % coverage of the collection filter by particulate. If the total suspended particulate is largely organic material, the limit of detection can be lowered significantly by using an indirect preparation method.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4225:1994, *Air quality — General aspects — Vocabulary.*

ISO 4226:1993, *Air quality — General aspects — Units of measurement.*

ISO Standard Handbook No. 2:1993, *Quantities and units.*

ISO Standard Handbook No. 3:1989, *Statistical Methods.*

3 Definitions

For the purposes of this International Standard, the following definitions apply (see also ISO 4225).

3.1 acicular: The shape of an extremely slender crystal with cross-sectional dimensions which are small relative to its length, i.e. needle-like.

3.2 amphibole: A group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, with the nominal formula:

$$A_0 \text{ or } {}_1B_2C_5T_8O_{22}(OH,F,Cl)_2$$

where

$A = K, Na$

$B = Fe^{2+}, Mn, Mg, Ca, Na$

$C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+}$

$T = Si, Al, Cr, Fe^{3+}, Ti$

In some varieties of amphibole, these elements can be partially substituted by Li, Pb or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124°.

3.3 amphibole asbestos: Amphibole in an asbestiform habit.

3.4 analytical sensitivity: The calculated airborne asbestos structure concentration in asbestos structures/litre, equivalent to counting of one asbestos structure in the analysis. The method in this International Standard does not specify an analytical sensitivity.

3.5 asbestiform: A specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility.

3.6 asbestos: A term applied to a group of silicate minerals belonging to the serpentine and amphibole groups which have crystallized in the asbestiform habit, causing them to be easily separated into long, thin, strong fibres when crushed or processed. The Chemical Abstracts Service Registry Numbers of the most common asbestos varieties are: chrysotile (12001-29-5), crocidolite (12001-28-4), grünerite asbestos (amosite) (12172-73-5), anthophyllite asbestos (77536-67-5), tremolite asbestos (77536-68-6) and actinolite asbestos (77536-66-4).

3.7 asbestos structure: A term applied to any connected or overlapping grouping of asbestos fibres or bundles, with or without other particles.

3.8 aspect ratio: The ratio of length to width of a particle.

3.9 blank: A structure count made on TEM specimens prepared from an unused filter, to determine the background measurement.

3.10 camera length: The equivalent projection length between the specimen and its electron diffraction pattern, in the absence of lens action.

3.11 chrysotile: A fibrous mineral of the serpentine group which has the nominal composition

$$Mg_3Si_2O_5(OH)_4$$

Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Minor substitution of magnesium by Al^{3+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+} and Co^{2+} may also be present. Chrysotile is the most prevalent type of asbestos.

3.12 cleavage: The breaking of a mineral along one of its crystallographic directions.

3.13 cleavage fragment: A fragment of a crystal that is bounded by cleavage faces.

3.14 cluster: A structure in which two or more fibres, or fibre bundles, are randomly oriented in a connected grouping.

3.15 d-spacing: The distance between identical adjacent and parallel planes of atoms in a crystal.

3.16 electron diffraction: A technique in electron microscopy by which the crystal structure of a specimen is examined.

3.17 electron scattering power: The extent to which a thin layer of substance scatters electrons from their original directions.

3.18 energy dispersive X-ray analysis: Measurement of the energies and intensities of X-rays by use of a solid state detector and multichannel analyser system.

3.19 eucentric: The condition when the area of interest of an object is placed on a tilting axis at the intersection of the electron beam with that axis and is in the plane of focus.

3.20 field blank: A filter cassette which has been taken to the sampling site, opened, and then closed. Such a filter is used to determine the background structure count for the measurement.

3.21 fibril: A single fibre of asbestos, which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances.

3.22 fibre: An elongated particle which has parallel or stepped sides. For the purposes of this International Standard, a fibre is defined to have an aspect ratio equal to or greater than 5:1 and a minimum length of 0,5 µm.

3.23 fibre bundle: A structure composed of parallel, smaller diameter fibres attached along their lengths. A fibre bundle may exhibit diverging fibres at one or both ends.

3.24 fibrous structure: A fibre, or connected grouping of fibres, with or without other particles.

3.25 habit: The characteristic crystal growth form, (or combination of these forms), of a mineral, including characteristic irregularities.

3.26 limit of detection: The calculated airborne asbestos structure concentration in structures per litre, equivalent to counting 2,99 asbestos structures in the analysis.

3.27 matrix: A structure in which one or more fibres, or fibre bundles, touch, are attached to, or partially concealed by, a single particle or connected group of nonfibrous particles.

3.28 Miller index: A set of either three or four integer numbers used to specify the orientation of a crystallographic plane in relation to the crystal axes.

3.29 PCM equivalent fibre: A fibre of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm.

3.30 PCM equivalent structure: A fibrous structure of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm.

3.31 primary structure: A fibrous structure that is a separate entity in the TEM image.

3.32 replication: A procedure in electron microscopy specimen preparation in which a thin copy, or replica, of a surface is made.

3.33 selected area electron diffraction: A technique in electron microscopy in which the crystal structure of a small area of a sample is examined.

3.34 serpentine: A group of common rock-forming minerals having the nominal formula

$Mg_3Si_2O_5(OH)_4$

3.35 structure: A single fibre, fibre bundle, cluster or matrix.

3.36 twinning: The occurrence of crystals of the same species joined together at a particular mutual orientation, such that the relative orientations are related by a definite law.

3.37 unopened fibre: An asbestos fibre bundle of large diameter which has not been separated into its constituent fibrils or fibres.

3.38 zone-axis: The line or crystallographic direction through the centre of a crystal which is parallel to the intersection edges of the crystal faces defining the crystal zone.

4 Principle

A sample of airborne particulate is collected by drawing a measured volume of air through either a capillary-pore polycarbonate membrane filter of maximum pore size 0,4 µm or a cellulose ester (either mixed esters of cellulose or cellulose nitrate) membrane filter of maximum pore size 0,45 µm by means of a battery-powered or mains-powered pump. TEM specimens are prepared from polycarbonate filters by applying a thin film of carbon to the filter surface by vacuum evaporation. Small areas are cut from the carbon-coated filter, supported on TEM specimen grids, and the filter medium is dissolved away by a solvent extraction procedure. This procedure leaves a thin film of carbon which bridges the openings in the TEM specimen grid, and which supports each particle from the original filter in its original position. Cellulose ester filters are chemically treated to collapse the pore structure of the filter, and the surface of the collapsed filter is then etched in an oxygen plasma to ensure that all particles are exposed. A thin film of carbon is evaporated onto the filter surface and small areas are cut from the filter. These sections are supported on TEM specimen grids and the filter medium is dissolved away by a solvent extraction procedure.

The TEM specimen grids from either preparation method are examined at both low and high magnifications to check that they are suitable for analysis before carrying out a quantitative structure count on randomly-selected grid openings. In the TEM analysis, electron diffraction (ED) is used to examine the crystal structure of a fibre, and its elemental composition is determined by energy dispersive X-ray analysis (EDXA). For a number of reasons, it is not possible to identify each fibre unequivocally, and fibres are classified according to the techniques which have been used to identify them. A simple code is used to record, for each fibre, the manner in which it was classified. The fibre classification procedure is based on successive inspection of the morphology, the electron diffraction pattern for a selected area, and the qualitative and quantitative energy dispersive X-ray analyses. Confirmation of the identification of chrysotile is done only by quantitative ED, and confirmation of amphibole is done only by quantitative EDXA and quantitative zone axis ED.

In addition to isolated fibres, ambient air samples often contain more complex aggregates of fibres, with or without other particles. Some particles are composites of asbestos fibres with other materials. Individual fibres and structures that are more complex are referred to as "asbestos structures". A coding system is used to record the type of fibrous structure, and to provide the optimum description of each of these complex structures. The two codes remove the requirement to interpret the structure counting data from the microscopist, and allow this evaluation to be made later without the requirement for re-examination of the TEM specimens. Several levels of analysis are specified, the higher levels providing a more rigorous approach to the identification of fibres. The procedure permits a minimum required fibre identification criterion to be defined on the basis of previous knowledge, or lack of it, about the particular sample. Attempts are then made to achieve this minimum criterion for each fibre, and the degree of success is recorded for each fibre. The lengths and widths of all classified structures and fibres are recorded. The number of asbestos structures found on a known area of the microscope sample, together with the equivalent volume of air filtered through this area, is used to calculate the airborne concentration in asbestos structures/litre of air.

5 Symbols of units and abbreviations

5.1 Symbols of units (see also ISO 4226 and ISO No. 2)

eV = electron volt

kV = kilovolt

l/min = litres per minute

µg = microgram (10^{-6} gram)

µm = micrometre (10^{-6} metre)

nm = nanometre (10^{-9} metre)

W = watt

5.2 Abbreviations

DMF Dimethylformamide

DE Electron diffraction

EDXA Energy dispersive X-ray analysis

FWHM Full width, half maximum

HEPA High efficiency particle absolute

MEC Mixed esters of cellulose

PC Polycarbonate

PCM Phase contrast optical microscopy

SAED Selected area electron diffraction

SEM Scanning electron microscope

STEM Scanning transmission electron microscop

TEM Transmission electron microscope

UICC Union Internationale Contre le Cancer

6 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water (6.1).

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

6.1 water, fibre-free.

A supply of freshly distilled, fibre-free water, or another source of fibre-free, pyrogen-free water shall be used.

6.2 Chloroform, analytical grade, distilled in glass, preserved with 1 % (*V/V*) ethanol.

6.3 1-Methyl-2-pyrrolidone.

6.4 Dimethylformamide.

6.5 Glacial acetic acid.

6.6 Acetone.

7 Apparatus

7.1 Air sampling — Equipment and consumable supplies

7.1.1 Filter cassette

Field monitors, comprising 25 mm to 50 mm diameter three-piece cassettes, with cowls which project less than 2 cm in front of the filter surface shall be used for sample collection. The cassette shall be loaded with either a capillary pore polycarbonate filter of maximum pore size 0,4 µm or an MEC or cellulose nitrate filter of maximum pore size 0,45 µm. Either type of filter shall be backed by a 5 µm pore size MEC or cellulose nitrate filter, and supported by a cellulose back-up pad. When the filters are in position, an elastic cellulose band or adhesive tape shall be applied to prevent air leakage. Suitable precautions shall be taken to ensure that the filters are tightly clamped in the assembly, so that significant air leakage around the filter cannot occur.

Representative filters from the filter lot shall be analysed as specified in 9.7 for the presence of asbestos structures before any are used for air sample collection.

7.1.2 Sampling pump

The sampling pump shall be capable of a flow-rate sufficient to achieve the desired analytical sensitivity. The face velocity through the filter shall be between 4,0 cm/s and 25,0 cm/s. The sampling pump used shall provide a non-fluctuating airflow through the filter, and shall maintain the initial volume flow-rate to within ± 10 % throughout the sampling period. A constant flow or critical orifice controlled pump meets these requirements. Flexible tubing shall be used to connect the filter cassette to the sampling pump. A means for calibration of the flow-rate of each pump is also required.

7.1.3 Stand

A stand shall be used to hold the filter cassette at the desired height for sampling, and shall be isolated from the vibrations of the pump (7.1.2).

7.1.4 Variable area flowmeter

A calibrated variable are a flowmeter with a range of approximately 1 l/min to 10 l/min is required for calibration of the air sampling system.

The variable area flowmeter shall be cleaned before use to avoid transfer of asbestos contamination from the flowmeter to the sample being collected.

7.2 Specimen preparation laboratory

Asbestos, particularly chrysotile, is present in varying quantities in many laboratory reagents. Many building materials also contain significant amounts of asbestos or other mineral fibres which may interfere with the analysis if they are inadvertently introduced during preparation of specimens. It is most important to ensure that, during preparation, contamination of TEM specimens by any extraneous asbestos fibres is minimized. All specimen preparation steps shall therefore be performed in an environment where contamination of the sample is minimized. The primary requirement of the sample preparation laboratory is that a blank determination shall yield a result which will meet the requirements specified in 9.7. A minimum facility considered suitable for preparation of TEM specimens is a laminar flow hood with positive pressure. However, it has been established that work practices in specimen preparation appear to be more important than the tape of clean handling facilities in use. Preparation of samples shall be carried out only after acceptable blank values have been demonstrated.

NOTE 1 It is recommended that activities involving manipulation of bulk asbestos samples not be performed in the same area as TEM specimen preparation, because of the possibilities of contaminating the TEM specimens.

7.3 Equipment for analysis

7.3.1 Transmission electron microscope

A TEM operating at an accelerating potential of 80 kV to 120 kV, with a resolution better than 1,0 nm, and a magnification range of approximately × 300 to × 100 000 shall be used. The ability to obtain a direct screen magnification of about × 100 000 is necessary for inspection of fibre morphology; this magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular if it cannot be obtained directly. It is also required that the viewing screen of the microscope be calibrated such that the lengths and widths of fibre images down to 1 mm width can be measured in increments of 1 mm, regardless of image orientation. This requirement is often fulfilled through the use of a fluorescent screen with calibrated gradations in the form of circles, as shown in figure 1.

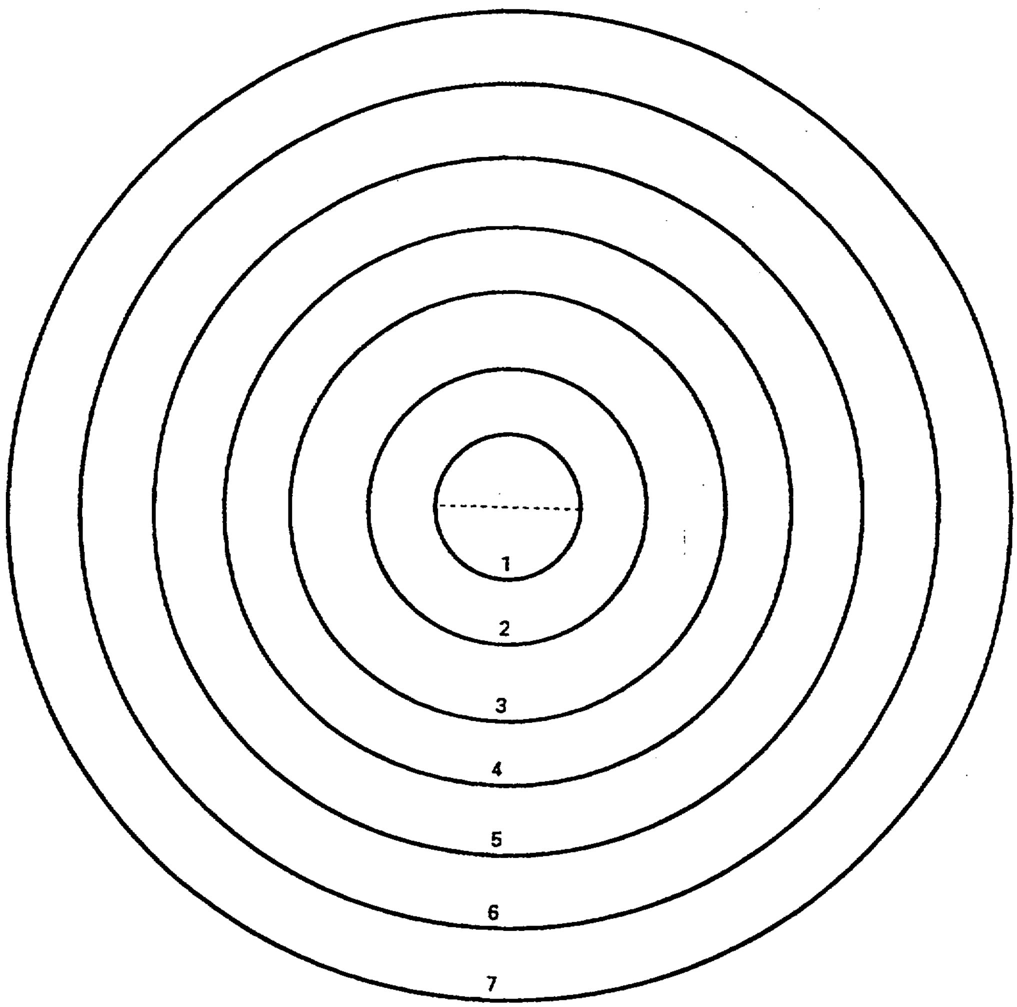


Figure 1 — Example of calibration markings on TEM viewing screen

For Bragg angles less than 0,01 rad, the TEM shall be capable of performing ED from an area of 0,6 μm^2 or less, selected from an in-focus image at a screen magnification of × 20 000. This performance requirement defines the minimum separation between particles at which independent ED patterns can be obtained from each particle. If SAED is used, the performance of a particular instrument may normally be calculated using the following equation

$$A = 0{,}785\ 4 \times \left(\frac{D}{M} + 2\ 000 C_s \theta^3 \right)^2$$

where

- A is the effective SAED area, in square micrometres;
- D is the diameter, in micrometres, of the SAED aperture;
- M is the magnification of the objective lens;
- C_s is the spherical aberration coefficient, in millimetres, of the objective lens;
- θ is the maximum required Bragg angle, in radians.

It is not possible to reduce the effective SAED area indefinitely by the use of progressively smaller SAED apertures, because there is a fundamental limitation imposed by the spherical aberration coefficient of the objective lens.

If zone-axis ED analyses are to be performed, the TEM shall incorporate a goniometer stage which permits the TEM specimen to be either

a) rotated through 360°, combined with tilting through at least + 30° to − 30° about an axis in the plane of the specimen;

b) tilted through at least + 30° to − 30° about two perpendicular axes in the plane of the specimen.

The analysis is greatly facilitated if the goniometer permits eucentric tilting, although this is not essential. If EDXA and zone-axis ED are required on the same fibre, the goniometer shall be of a type which permits tilting of the specimen and acquisition of EDXA spectra without changing the specimen holder.

The TEM shall have an illumination and condenser lens system capable of forming an electron probe of diameter less than 250 nm.

NOTE 2 Use of an anti-contamination trap around the specimen is recommended if the required instrumental performance is to be obtained.

7.3.2 Energy dispersive X-ray analyser

The TEM shall be equipped with an energy dispersive X-ray analyser capable of achieving a resolution better than 180 eV (FWHM) on the MnKα. Since the performance of individual combinations of TEM and EDXA equipment is dependent on a number of geometrical factors, the required performance of the combination of the TEM and X-ray analyser is specified in terms of the measured X-ray intensity obtained from a fibre of small diameter, using a known electron beam diameter. Solid state X-ray detectors are least sensitive in the low energy region, and so measurement of sodium in crocidolite shall be the performance criterion. The combination of electron microscope and X-ray analyser shall yield, under routine analytical conditions, a background-subtracted NaKα integrated peak count rate of more than 1 count per second (cps) from a fibre of UICC crocidolite, 50 nm in diameter or smaller, when irradiated by an electron probe of 250 nm diameter or smaller at an accelerating potential of 80 kV. The peak/background ratio for this performance test shall exceed 1,0.

The EDXA unit shall provide the means for subtraction of the background, identification of elemental peaks, and calculation of background-subtracted peak areas.

7.3.3 Computer

Many repetitive numerical calculations are necessary, and these may be performed conveniently by relatively simple computer programmes. For analyses of zone-axis ED pattern measurements, a computer with adequate memory is required to accommodate the more complex programmes involved.

7.3.4 Plasma asher

For preparation of TEM specimens from MEC filters, a plasma asher, with a radio frequency power rating of 50 W or higher, shall be used to etch the surface of collapsed MEC filters. The asher shall be supplied with a controlled oxygen flow, and shall be modified, if necessary, to provide a valve to control the speed of air admission so that rapid air admission does not disturb particulates from the surface of the filter after the etching step.

NOTE 3 It is recommended that filters be fitted to the oxygen supply and the air admission line.

7.3.5 Vacuum coating unit

A vacuum coating unit capable of producing a vacuum better than 0,013 Pa shall be used for vacuum deposition of carbon on the membrane filters. A sample holder is required which will allow a glass microscope slide to be continuously rotated during the coating procedure.

NOTE 4 A mechanism which also allows the rotating slide to be tilted through an angle of approximately 45° during the coating procedure is recommended. A liquid nitrogen cold trap above the diffusion pump may be used to minimize the possibility of contamination of the filter surfaces by oil from the pumping system. The vacuum coating unit may also be used for deposition of the thin film of gold, or other calibration material, when it is required on TEM specimens as an internal calibration of ED patterns.

7.3.6 Sputter coater

A sputter coater with a gold target may be used for deposition of gold onto TEM specimens as an integral calibration of ED patterns. Other calibration materials are acceptable. Experience has shown that a sputter coater allows better control of the thickness of the calibration material.

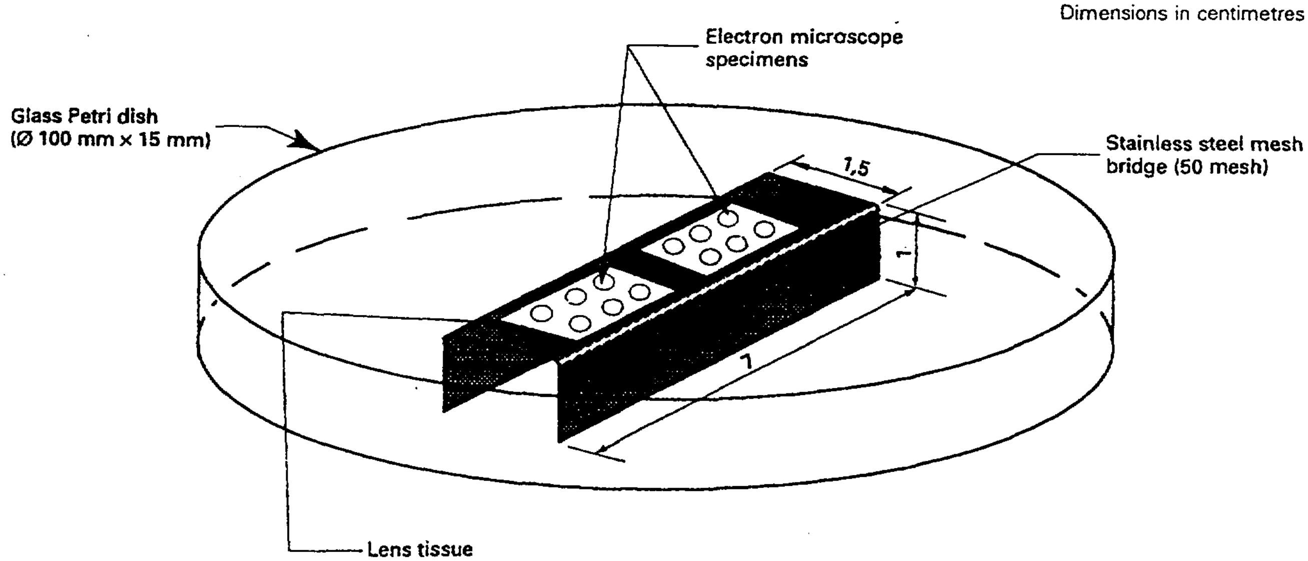
7.3.7 Solvent washer (Jaffe washer)

The purpose of the Jaffe washer is to allow dissolution of the filter polymer while leaving an intact evaporated carbon film supporting the fibres and other particles from the filter surface. One design of a washer which has been found satisfactory for various solvents and filter media is shown in figure 2. In general, either chloroform or 1-methyl-2-pyrrolidone has been used for dissolving polycarbonate filters and dimethylformamide or acetone has been used for dissolving MEC or cellulose nitrate filters. The higher evaporation rates of chloroform and acetone require that a reservoir of 10 ml to 50 ml of solvent be used, which may need replenishment during the procedure. Dimethylformamide and 1-methyl-2-pyrrolidone have lower vapour pressures and much smaller volumes of solvent may be used. It is recommended that all washers be used in a fume hood, and when specimens are not being inserted or removed, the Petri dish lid shall be in place during the solvent dissolution. The washer shall be cleaned before it is used for each batch of specimens.

7.3.8 Condensation washer

For more rapid dissolution of the filter polymer, or if difficulties are experienced in dissolving the filter polymer, use a condensation washer, consisting of a flask, condenser and cold finger assembly, with a heating mantle and means for controlling the temperature. A suitable assembly is shown in figure 3, using either acetone or chloroform as the solvent, depending on the type of filter.

Dimensions in centimetres



NOTE — Solvent is added until the meniscus contacts the underside of the stainless steel mesh bridge.

Figure 2 — Example of design of solvent washer (Jaffe washer)

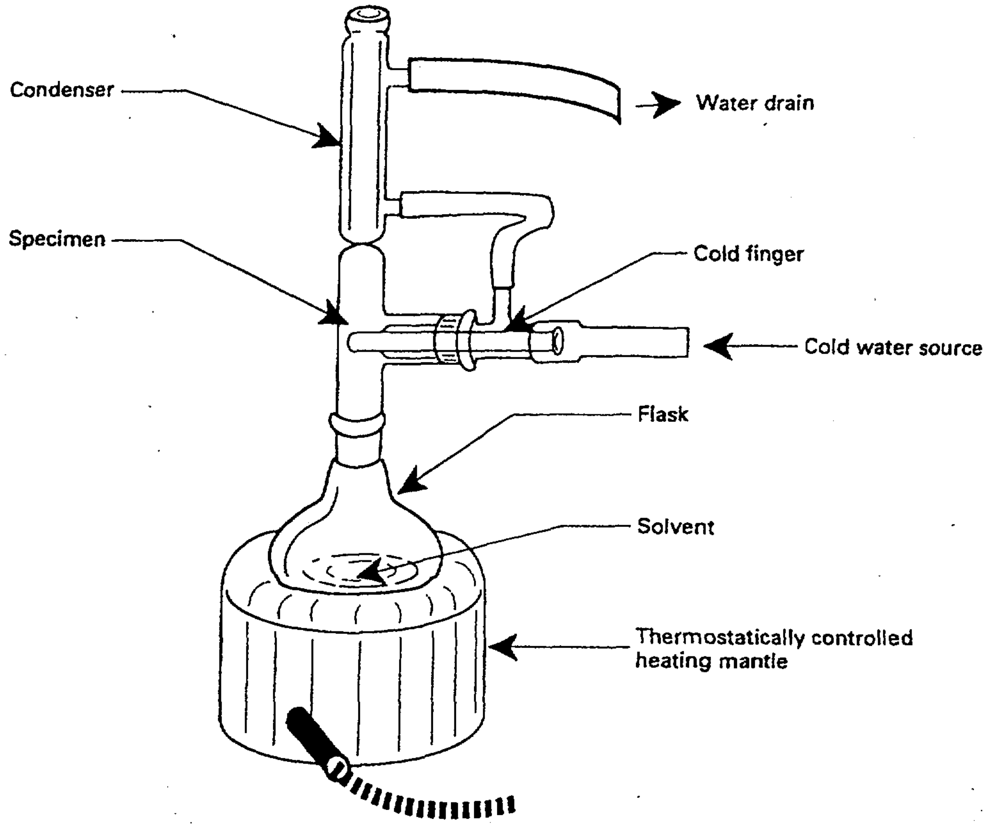


Figure 3 — Example of design of condensation washer

7.3.9 Slide warmer or oven

Use either a slide warmer or an oven for heating slides during the preparation of TEM specimens from MEC or cellulose nitrate filters. It is required to maintain a temperature of 65 °C to 70 °C.

7.3.10 Ultrasonic bath

An ultrasonic bath is necessary for cleaning the apparatus used for TEM specimen preparation.

7.3.11 Carbon grating replica

A carbon grating replica with about 2 000 parallel lines per millimetre shall be used to calibrate the magnification of the TEM.

7.3.12 Calibration specimen grids for EDXA

TEM specimen grids prepared from dispersions of calibration minerals are required for calibration of the EDXA system. Some suitable calibration minerals are riebeckite, chrysotile, halloysite, phlogopite, wollastonite and bustamite. The mineral used for calibration of the EDXA system for sodium shall be prepared using a gold TEM grid.

7.3.13 Carbon rod sharpener

The use of necked carbon rods, or equivalent, allows the carbon to be evaporated onto the filters with a minimum of heating.

7.3.14 Disposable tip micropipettes

A disposable tip micropipette, capable of transferring a volume of approximately 30 µl, is necessary for the preparation of TEM specimen grids from MEC filters.

7.4 Consumable supplies

7.4.1 Copper electron microscope grids

Copper TEM grids with 200 mesh are recommended. Grids which have grid openings of uniform size such that they meet the requirement specified in 9.6.2 shall be chosen. To facilitate the relocation of individual grid openings for quality assurance purposes, the use of grids with numerical or alphabetical indexing of individual grid openings is recommended.

7.4.2 Gold electron microscope grids

Gold TEM grids with 200 mesh are recommended to mount TEM specimens when sodium measurements are required in the fibre identification procedure. Grids which have grid openings of uniform size such that they meet the requirement specified in 9.6.2 shall be chosen. To facilitate the relocation of individual grid openings for quality assurance purposes, the use of grids with numerical or alphabetical indexing of individual grid openings is recommended.

7.4.3 Carbon rod electrodes

Spectrochemically pure carbon rods, shall be used in the vacuum evaporator (7.3.5) during carbon coating of filters.

7.4.4 Routine electron microscopy tools and supplies

Fine-point tweezers, scalpel holders and blades, microscope slides, double-coated adhesive tape, lens tissue, gold wire, tungsten filaments and other routine supplies are required.

7.4.5 Reference asbestos samples

Asbestos samples, shall be for preparation of reference TEM specimens of the primary asbestos minerals. The UICC set of minerals is suitable for this purpose.

8 Air sample collection

The desired analytical sensitivity is a parameter that shall be established for the analysis prior to sample collection. It is defined as the structure concentration corresponding to the detection of one structure in the analysis. For direct transfer methods of TEM specimen preparation, the analytical sensitivity is a function of the volume of air sampled, the active area of the collection filter, and the area of the TEM specimen over which structures are counted. If total airborne dust levels are high, it may be necessary to terminate sampling before the required volume has been sampled. If this happens, the analytical sensitivity required can be achieved only by counting structures on more grid openings, or by selective concentration of asbestos structures using an indirect TEM specimen preparation technique. Select the sampling rate and the period of sampling to yield the required analytical sensitivity, as detailed in table 1. Before air samples are collected, unused filters shall be analysed as described in 9.7 to determine the mean asbestos structure count for blank filters.

Air samples shall be collected using filter cassettes (7.1.1). During sampling, the cassette shall be supported on a stand (7.1.3) which is isolated from the vibrations of the pump (7.1.2). The cassette shall be held facing vertically downwards at a height of approximately 1,5 m to 2,0 m above ground/floor level, and shall be connected to the pump with a flexible tube.

Measure the sampling flow-rate at the front end of the cassette, both at the beginning and end of the sampling period, using a calibrated variable area flowmeter (7.1.4) temporarily attached to the inlet of the cassette. The mean value of these two measurements shall be used to calculate the total air volume sampled.

Basic strategies for monitoring environmental sources of airborne asbestos are described in annex G. After sampling, a cap shall be placed over the open end of the cassette, and the cassette packed with the filter face-upwards for return to the laboratory. Field blank filters shall also be included, as specified in 9.7, and submitted to the remaining analytical procedures along with the samples.

NOTES

5 In table 1 a collection filter area of 385 mm^2 is assumed, and the TEM grid openings are assumed to be 85 μm^2 square. The limit of detection is defined as the upper 95 % confidence limit of the Poisson distribution for a count of 0 structures. In the absence of background, this is equal to 2,99 times the analytical sensitivity. Backgrounds that are different from 0 observed during analysis of blank filters will degrade the limit of detection.

6 The analytical sensitivity S, expressed in number of structures per litre, is calculated using the following equation:

$$S = \frac{A_f}{kA_gV}$$

where

- A_f is the active area, in square millimetres, of sample collection filter;
- A_g is the mean area, in square millimetres, of grid openings examined;
- k is the number of grid openings examined;
- V is the volume of air sampled, in litres.

Table 1 — Examples of the minimum number of grid openings required to achieve a particular analytical sensitivity and limit of detection

Analytical sensitivity structures/l	Limit of detection structures/l	Volume of air sampled (litres)					
		500	1 000	2 000	3 000	4 000	5 000
0,1	0,30	1 066	533	267	178	134	107
0,2	0,60	533	267	134	89	67	54
0,3	0,90	356	178	89	60	45	36
0,4	1,2	267	134	67	45	34	27
0,5	1,5	214	107	54	36	27	22
0,7	2,1	153	77	39	26	20	16
1,0	3,0	107	54	27	18	14	11
2,0	6,0	54	27	14	9	7	6
3,0	9,0	36	18	9	6	5	4
4,0	12	27	14	7	5	4	4
5,0	15	22	11	6	4	4	4
7,0	21	16	8	4	4	4	4
10	30	11	6	4	4	4	4

9 Procedure for analysis

9.1 General

The techniques used to prepare TEM specimens are different for polycarbonate and cellulose ester filters. The preparation method to be used shall be either 9.3 or 9.4, depending on the type of membrane filter used for air sampling. Cleaning of the sample cassettes before they are opened, preparation of the carbon evaporator, criteria for acceptable specimen grids, and the requirement for blank determinations are identical for the two preparation techniques. TEM examination, structure counting, fibre identification and reporting of results are independent of the type of filter or preparation technique used.

The ability to meet the blank sample criteria is dependent on the cleanliness of equipment and supplies. Consider all supplies such as microscope slides and glassware as potential sources of asbestos contamination. It is necessary to wash all glassware before it is used. Wash any tools or glassware which come into contact with the air sampling filters or TEM specimen preparations both before use and between handling of individual samples. Where possible, disposable supplies should be used.

9.2 Cleaning of sample cassettes

Asbestos fibres can adher to the exterior surfaces of air sampling cassettes, and these fibres can be inadvertently transferred to the sample during handling. To prevent this possibility of contamination, and after ensuring that the cassette is tightly sealed, wipe the exterior surfaces of each sampling cassette before it is placed in the clean facility or laminar flow hood.

9.3 Direct preparation of TEM specimens from polycarbonate filters

9.3.1 Selection of filter area for carbon coating

Use a cleaned microscope slide to support representative portions of polycarbonate filter during the carbon evaporation. Double-coated adhesive tape is used to attach the filter portions to the glass slide. Take care not to stretch the polycarbonate filters during handling. Using freshly cleaned tweezers, remove the polycarbonate filter from the sampling cassette, and place it on to a second cleaned glass microscope slide which is used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut the filter by rocking the blade from the point, pressing it into contact with the filter. Repeat the process as necessary. Several such portions may be mounted on the same microscope slide. The scalpel blade and tweezers shall be washed and dried between the handling of each filter. Identify the filter portions by writing on the glass slide.

9.3.2 Carbon coating of filter portions

Place the glass slide holding the filter portions on the rotation-tilting device, approximately 10 cm to 12 cm from the evaporation source, and evacuate the evaporator chamber (7.3.5) to a vacuum better than 0,013 Pa. The evaporation of carbon shall be performed in very short bursts, separated by a few seconds to allow the electrodes to cool. If evaporation of carbon is too rapid, the strips of polycarbonate filter will begin to curl, and cross-linking of the surface will occur. This cross-linking procedures a layer of polymer which is relatively insoluble in organic solvents, and it will not be possible to prepare satisfactory TEM specimens. The thickness of carbon required is dependent on the size of particles on the filter, and approximately 30 nm to 50 nm has been found to be satisfactory. If the carbon film is too thin, large particles will break out of the film during the later stages of preparation, and there will be few complete and undamaged grid openings on the specimen. Too thick a carbon film will lead to a TEM image which is lacking in contrast, and the ability to obtain ED patterns will be compromised. The carbon film thickness should be the minimum possible, while retaining most of the grid openings of the TEM specimen intact.

9.3.3 Preparation of the Jaffe washer

Place several pieces of lens tissue, as shown in figure 2, on the stainless steel bridge (7.1.3) and fill the washer (see 7.3.7) with chloroform (6.2) or 1-methyl-2-pyrrolidone (6.3) to a level where the meniscus contacts the underside of the mesh, resulting in saturation of the lens tissue.

9.3.4 Placing of specimens in the Jaffe washer

Using a curved scalpel blade, cut three 3 mm square pieces of carbon-coated polycarbonate filter form the carbon-coated filter portion. Select three squares to represent the centre and the periphery of the active surface of the filter. Place each square of filter, carbon side up, on a TEM specimen grid, and place the grid and filter on the saturated lens tissue in the Jaffe washer. Place the three specimen grids from one sample on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. Cover the Jaffe washer with the lid, and allow the washer to stand for at least 8 h.

NOTE 7 It has been found that some polycarbonate filters will not completely dissolve in the Jaffe washer, even after exposure to chloroform for as long as 3 d. This problem is more severe if the surface of the filter was overheated during the carbon evaporation. It has been found that the problem of residual undissolved filter polymer can be overcome in several ways:

a) condensation washing of the grids, using chloroform as the solvent, after the initial Jaffe washer treatment, can often remove much of the residual filter medium in a period of approximately 30 min. To carry out this procedure, transfer the piece of lens tissue supporting the specimen grids to the cold finger of the condensation washer (7.3.8), which has achieved stable operating conditions. Operate the washer for approximately 30 min after inserting the grids;

b) used in a Jaffe washer, 1-methyl-2-pyrrolidone has been found to be a more effective solvent than chloroform for polycarbonate filters. This solvent is more effective if the lens paper is not used and grids are placed directly on the stainless steel mesh of the Jaffe washer. A dissolution period of 2 h to 6 h has been found to be satisfactory. After dissolution is complete, remove the stainless steel mesh from the Jaffe washer and allow the grids to dry. 1-methyl-2-pyrrolidone evaporates very slowly. If it is required to dry the grids more rapidly, transfer the stainless steel bridge into another Petri dish, and add water (6.1) until the meniscus contacts the underside of the mesh. After approximately 15 min, remove the mesh and allow the grids to dry. If it is desired to retain water-soluble particle species on the TEM grids, ethanol may be used instead of water (6.1) for the second wash;

c) a mixture of 20 % 1,2-diaminoethane [ethylenediamine] and 80 % 1-methyl-2-pyrrolidone, used in a Jaffe washer, completely dissolves polycarbonate filters in 15 min, even if the surface of the filter has been overheated. To use this solvent place the grids directly on the stainless steel mesh of the Jaffe washer, do not use the lens paper. After a period of 15 min, transfer the stainless steel bridge into another Petri dish, and add water (6.1) until the meniscus contacts the underside of the mesh. After approximately 15 min, remove the mesh and allow the grids to dry. If it is desired to retain water-soluble particle species on the TEM grids, ethanol may be used instead of water (6.1) for the second wash.

9.3.5 Rapid preparation of TEM specimens from PC filters

TEM specimens can be prepared rapidly from PC filters, if desired, by washing for approximately 1 h in a Jaffe washer, followed by washing for 30 min in a condensation washer using chloroform as the solvent. The alternative filter dissolution procedures described in note 7 may also be used.

9.4 Direct preparation of TEM specimens from cellulose ester filters

9.4.1 Selection of area of filter for preparation

Using clean tweezers, remove the filter from the filter cassette, and place it on a cleaned microscope slide. Using a clean, curved scalpel blade, cut out a portion of the filter.

9.4.2 Preparation of solution for collapsing cellulose ester filters

Mix 35 ml of dimethylformamide (6.4), and 15 ml of glacial acetic acid (6.5) with 50 ml of water (6.1). Store this mixture in a clean bottle. The mixture is stable and suitable for use for up to 3 months after preparation.

9.4.3 Filter collapsing procedure

Using a micropipette with a disposable tip (7.3.14), place 15 µl/cm^2 to 25 µl/cm^2 of the solution prepared in 9.4.2 on a cleaned microscope slide, and using the end of the pipette tip, spread the liquid over the area to be occupied by the filter portion. Place the filter portion, active surface upwards, on top of the solution, lowering the edge of the filter at an angle of about 20° so that air bubbles are not created. Remove any solution not absorbed by the filter by allowing a paper tissue to contact the liquid at the edge of the filter. More than one filter portion may be placed on one slide. Place the slide either on a thermostatically controlled slide warmer (7.3.9) at a temperature of 65 °C to 70 °C, or in an oven (7.3.9) at this temperature, for 10 min. The filter collapses slowly to about 15 % of its original thickness. The procedure leaves a thin, transparent polymer film, with particles and fibres embedded in the upper surface.

9.4.4 Plasma etching of the filter surface

The optimum conditions and time for plasma etching (see 7.3.4) have been determined experimentally from the recovery of fine chrysotile fibrils on 0,8 µm pore size MEC filters. The conditions required in a particular plasma asher shall be established using the procedure specified in annex A. Place the microscope slide holding the collapsed filter portions in the plasma asher, and etch for the time and under the conditions determined. Take care to ensure that the correct conditions are respected. After etching, admit air slowly to the chamber and remove the microscope slide.

Adjust the air admission valve of the plasma asher such that the time taken for the chamber to reach atmospheric pressure exceeds 2 min. Rapid air admission may disturb particulates on the surface of the etched filter.

9.4.5 Carbon coating

Coat the microscope slide holding the collapsed filter portions with carbon as specified in 9.3.2.

9.4.6 Preparation of the Jaffe washer

Place several pieces of lens tissue on the stainless steel bridge, and fill the washer with dimethylformamide (6.4) or acetone (6.6) to a level where the meniscus contacts the underside of the mesh, resulting in saturation of the lens tissue.

9.4.7 Placing of specimens in the Jaffe washer

Place the specimens in the Jaffe washer as specified in 9.3.4. Specimens are normally cleared after approximately 4 h.

9.4.8 Rapid preparation of TEM specimens from cellulose ester filters

An alternative washing procedure may be used to prepare TEM specimens from cellulose ester filters more rapidly than can be achieved by the Jaffe washing procedure. After the specimens have been washed in a Jaffe washer for approximately 1 h, transfer the piece of lens tissue supporting the specimens to the cold finger of a condensation washer (7.3.8) operating with acetone as the solvent because dimethylformamide shall not be used in a condensation washer. Operate the condensation washer for approximately 30 min. This treatment removes all the remaining filter polymer.

9.5 Criteria for acceptable TEM specimen grids

Valid data cannot be obtained unless the TEM specimens meet specified quality criteria. Examine the TEM specimen grid in the electron microscope at a sufficiently low magnification (× 300 to × 1 000) for complete grid openings to be inspected. Reject the grid if

a) the TEM specimen has not been cleared of filter medium by the filter dissolution step. If the TEM specimen exhibits areas of undissolved filter medium, and if at least two of the three specimen grids are not cleared, either additional washing with solvent shall be carried out, or new specimens shall be prepared from the filter;

b) the sample is overloaded with particulate. If the specimen grid exhibits more than approximately 10 % obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. This filter cannot be alanysed satisfactorily using the direct preparation methods because the grid is too heavily loaded with debris to allow separate examination of individual particles by ED and EDXA, and obscuration of fibres by other particulates may lead to underestimation of the asbestos structure count;

c) the particulate deposits on the specimen are not uniformly distributed from one grid opening to the next. If the particulate deposits on the specimen are obviously not uniform from one grid opening to the next, the specimen shall be designated as non-uniform. This condition is a function either of the air sampling conditions or of the fundamental nature of the airborne particulate. Satisfactory analysis of this filter may not be possible unless a large number of grid openings is examined;

d) the TEM grid is too heavily loaded with fibrous structures to make an accurate count. Accurate counts cannot be made if the grid has more than approximately 7 000 structures/mm^2; or

e) more than approximately 25 % of the grid openings have broken carbon film over the whole grid opening. Since the breakage of carbon film is usually more frequent in areas of heavy deposit, counting of the intact openings can lead to an underestimate of the asbestos structure count.

NOTE 8 If the specimens are rejected because unacceptable numbers of grid openings exhibit broken carbon replica, an additional carbon coating may be applied to the carbon coated filter, and new specimen grids prepared. The larger particles can often be supported by using a thicker carbon film. If this action does not produce acceptable specimen grids, this filter cannot be analysed using the direct preparation methods.

If one or more of the conditions described in b), c), d) or e) exists, it may not be possible to analyse the sample by this method.

9.6 Procedure for structure counting by TEM

9.6.1 General

The examination consists of a count of asbestos structures which are present on a specified number of grid openings. Fibres are classified into groups on the basis of morphological observations, ED patterns and EDXA spectra. The total number of structures to be counted depends on the statistical precision desired. In the absence of asbestos structures, the area of the TEM specimen grids which must be examined depends on the analytical sensitivity required. The precision of the structure count depends not only on the total number of structures counted, but also on their uniformity from one grid opening to the next. Additional structure counting will be necessary if greater precision is required.

In order that the estimate of the structure density on the sampling filter shall not be based on the small area represented by one specimen grid, grid openings shall be examined on two of the three specimen grids prepared. Then combine the results in the calculation of the structure density. Structure counts shall be made at a magnification of approximately × 20 000, and shall be terminated at the end of the examination of the grid opening on which the 100th asbestos structure is observed, except that the count shall be continued until a minimum of 4 grid openings have been examined. Otherwise, the structure count shall continue to that number of grid openings at which the specified analytical sensitivity has been achieved.

NOTE 9 The normal range for the number of grid openings which should be examined is from 4 to 20. If insufficient air has been sampled through the filter, the calculation in 9.6.4 can indicate that an impractically large number of grid openings should be examined. When this situation occurs, a larger value of analytical sensitivity may have to be accepted.

9.6.2 Measurement of mean grid opening area

The mean grid opening area shall be measured for the type of TEM specimen grids in use. The standard deviation of the mean of 10 openings selected from 10 grids should be less than 5 %. As an optional procedure, or if the 5 % standard deviation criterion cannot be demonstrated, the dimensions of each grid opening examined in the TEM shall be measured at a calibrated magnification.

9.6.3 TEM alignment and calibration procedures

Before structure counting is performed, align the TEM according to instrumental specifications. Calibrate the TEM and EDXA system according to the procedures specified in annex B.

9.6.4 Determination of stopping point

Before structure counting is begun, calculate the area of specimen to be examined in order to achieve the selected analytical sensitivity. Calculate the maximum number of grid openings to be examined using the following equation:

$$k = \frac{A_f}{A_g V S}$$

where

k is the number of grid openings to be examined, rounded upwards to the next highest integer;

A_f is the area, in square millimetres, of sample filter;

A_g is the area, in square millimetres, of TEM specimen grid opening;

V is the volume of air sampled, in litres;

S is the required analytical sensitivity, expressed in number of structures per litre.

9.6.5 General procedure for structure counting and size analysis

Use at least two specimen grids prepared from the filter in the structure count. Select at random several grid openings from each grid, and combine the data in the calculation of the results.

Use a form similar to that shown in figure 4 to record the data. Insert the first specimen grid into the TEM.

NOTE 10 In order to facilitate quality assurance measurements which require re-examination of the same grid opening by different microscopists, the grid should be inserted into the specimen holder in a standard orientation with the grid bars parallel and perpendicular to the axis of the specimen holder. This will provide scan directions parallel to the edges of the grid opening. It should be ensured that all microscopists begin scanning at the same starting point on the grid opening, and that they use similar scan patterns. This procedure permits rapid relocation of fibrous structures for further examination if necessary.

Select a typical grid opening and set the screen magnification to the calibrated value (approximately × 20 000). Adjust the sample height until the features in the centre of the TEM viewing screen are at the eucentric point. Set the goniometer tilt angle to zero. In column 1 of the data recording form, record the number or letter used to identify the grid. In column 2, record the identification of the particular grid opening. Position the specimen so that the grid opening is positioned with one corner visible on the screen. Move the image by adjustment of only one translation control, carefully examining the sample for fibres, until the opposite side of the grid opening is encountered. Move the image by a predetermined distance less than one screen diameter, using the other translation control, and scan the image in the reverse direction. Continue the procedure in this manner until the entire grid opening has been inspected in a pattern similar to that shown in figure 5. When a fibrous structure is detected, assign a sequential number to the primary structure in column 3, perform the identification procedures required as detailed in annex E, and enter the appropriate compositional classification on the structure counting form in column 5. Assign a morphological classification to the structure according to the procedures specified in annex D, and record this in column 6. Measure on the TEM viewing screen the length and width of the image of the primary structure, in millimetres, and record these measurements in columns 7 and 8. For a disperse cluster or matrix, assign a compositional classification and a morphological classification to each structure component, measure the length and width, and enter the data in columns 4 to 8. Use column 4 of the data recording form to tabulate the sequential number of total structures taking into account structure components. if non-asbestos fibres are observed, note their presence and type, if known. After a fibrous structure has been examined and measured, relocate the original field of view accurately before continuing scanning of the specimen. Failure to do this may cause structures to be overlooked or counted twice. Continue the examination until the completion of the grid opening on which the 100th asbestos structure has been recorded, or until the number of grid openings required to achieve the specified analytical sensitivity, calculated according to 9.6.4, have been examined whichever occurs first. The data shall be drawn approximately equally from a minimum of two grids. Regardless of the value calculated according to 9.6.4, fibrous structures on a minimum of four openings shall be counted.

9.6.6 Measurement of concentration for asbestos fibres and bundles longer than 5 µm

Consider improving the statistical validity for measurement of asbestos fibres and bundles longer than 5 µm by additional examination at a lower magnification, taking account only of the longer fibres and bundles. Perform this extended examination for fibres and bundles longer than 5 µm in accordance with the procedures specified in annex E. Use a magnification of approximately × 10 000 for counting all asbestos fibres and bundles longer than 5 µm, or approximately × 5 000 if only fibres and bundles within the diameter range 0,2 µm to 3,0 µm are to be counted. Continue the count until completion of the grid opening on which 100 fibres and bundles have been recorded, or until a sufficient area of the specimen has been examined to achieve the desired analytical sensitivity. Only those structures which are identified as, or are suspected to be, either chrysotile or one of the amphibole minerals will be reported in either the original or the extended TEM examination. Other materials, such as gypsum, cellulose fibres, and filter artifacts such as undissolved filter strands, will not be included in the fibre count. This restriction is intended to ensure that the best statistical validity is obtained for the materials of interest.

TEM asbestos structure count (page of)

Report number: ....
Sample number: ....
File name: ....
Sample description: ....
....
....
Preparation date: .... By: ....
Analysis date: .... By: ....
Computer entry date: .... By: ....

Air volume: .... litres

Sample filter area: .... mm^2

Magnification: ....

Grid opening dimension: .... µm

Level of analysis (C): ....
(A): ....

Grid	Grid opening	Number of structures		Class	Type of structure	Length	Width	Comments
		primary	total			mm	mm	

Figure 4 — Example of structure counting form

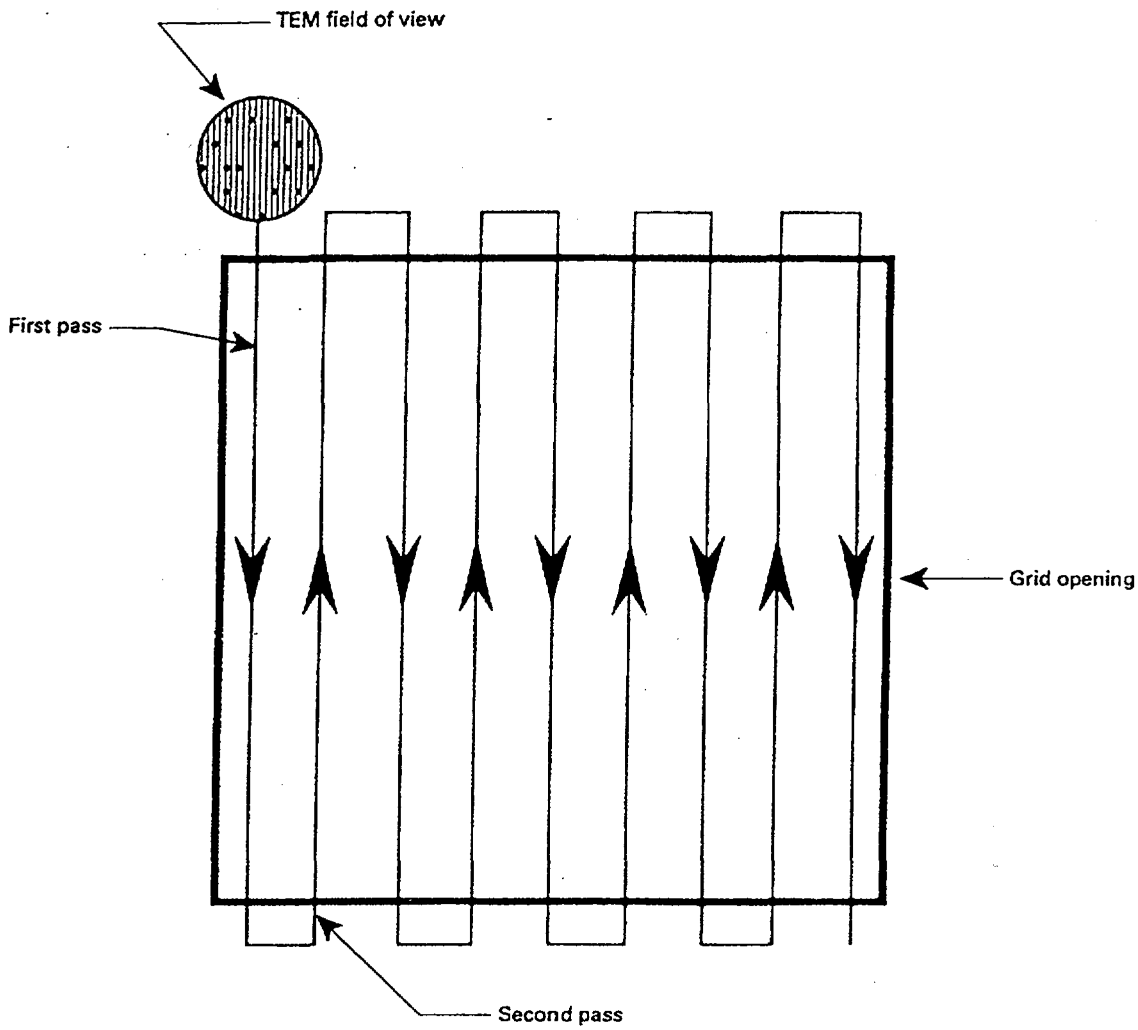


Figure 5 — Example of scanning procedure for TEM specimen examination

9.7 Blank and quality control determinations

Before air samples are collected, a minimum of two unused filters from each filter lot of 100 filters shall be analysed to determine the mean asbestos structure count. If the mean count for all types of asbestos structures is found to be more than 10 structures/mm^2, or if the mean fibre count for asbestos fibres and bundles longer than 5 µm is more than 0,1 fibre/mm^2, reject the filter lot.

To ensure that contamination by extraneous asbestos fibres during specimen preparation is insignificant compared with the results reported on samples, establish a continuous programme of blank measurements. At least one field blank shall be processed along with each batch of samples. In addition, at least one unused filter shall be included with every group of samples prepared on one microscope slide.

Initially, and also at intervals afterwards, ensure that samples of known asbestos concentrations can be analysed satisfactorily. Since there is a subjective component in the structure counting procedure, it is necessary that recounts of some specimens be made by different microscopists, in order to minimize the subjective effects. Such recounts provide a means of maintaining comparability between counts made by different microscopists. Variability between and within microscopists and between laboratories shall be characterized. These quality assurance measurements shall constitute approximately 10 % of the analyses. Repeat results should not differ at the 5 % significance level.

9.8 Calculation of results

Calculate the results using the procedures detailed in annex F. Prior to the TEM examination of the specimens, the level of analysis was specified. Before the results are calculated, the compositional and morphological classifications to be included in the result shall be specified. The chi-squared uniformity test shall be conducted using the number of primary asbestos structures found on each grid opening, prior to the application of the cluster and matrix counting criteria. The concentration result shall be calculated using the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

10 Performance characteristics

10.1 General

It is important to use this analytical method in conjunction with a continuous quality control programme. The quality control programme should include use of standard samples, blank samples, and both interlaboratory and intralaboratory analyses.

10.2 Interferences and limitations of fibre identification

Unequivocal identification of every chrysotile fibre is not possible, due to both instrumental limitations and the nature of some of the fibres. The requirement for a calibrated ED pattern eliminates the possibility of an incorrect identification of the fibre selected. However, there is a possibility of misidentification of fibres for which both the morphologies and the ED patterns are reported on the basis of visual inspection only. The only significant possibilities of misidentification occur with halloysite, vermiculite scrolls or palygorskite, all of which can be discriminated from chrysotile by the use of EDXA and by observation of the 0,73 nm (002) reflection of chrysotile in the ED pattern.

As in the case of chrysotile fibres, complete identification of every amphibole fibre is not possible due to instrumental limitations and the nature of some of the fibres. Moreover, complete identification of every amphibole fibre is not practical due to the limitations of both time and cost. Particles of a number of other minerals with compositions similar to those of some amphiboles could be erroneously classified as amphibole when the classification criteria do not include zone-axis ED techniques. However, the requirement for quantitative EDXA measurements on all fibres as support for the random orientation ED technique makes misidentification very unlikely, particularly when other similar fibres in the same sample have been identified as amphibole by zone-axis methods. The possibility of misidentification is further reduced with increasing aspect ratio, since it is rare for the minerals with which amphibole may be confused to display an asbestiform habit.

10.3 Precision and accuracy (see ISO Standard Handbook No. 3)

10.3.1 Precision

The analytical precision that can be obtained is dependent upon the number of structures counted, and also on the uniformity of the particulate deposit on the original filter. Assuming that the structures are randomly deposited on the filter, if 100 structures are counted and the loading is at least 3,5 structures/grid opening, computer modelling of the counting procedure shows that a coefficient of variation of about 10 % can be expected. As the number of structures counted decreases, the precision will also decrease approximately as $\sqrt{N}$, where N is the number of structures counted. In practice, particulate deposits obtained by filtration of ambient air samples are rarely ideally distributed, and it is found that the precision is correspondingly reduced. Degradation of precision is a consequence of several factors, such as:

a) non-uniformity of the filtered particulate deposit;

b) distorsion of the fibre distribution by application of the structure counting criteria;

c) variation between microscopists in their interpretation of the fibrous structures;

d) variation between microscopists in their ability to detect and identify fibres.

The 95 % confidence interval about the mean for a single structure concentration measurement using this analytical method should be approximately ± 25 % when 100 structures are counted over 10 grid openings.

10.3.2 Accuracy

There is no independent method available to determine the accuracy.

NOTE 11 It has been demonstrated that, after polycarbonate membrane filters have been coated with carbon, particulate material is transferred to the TEM specimens without measurable losses. However, if the filters are heavily loaded by particulate material, some of this may be lost before they are coated with carbon. Good comparability between the capillary-pore polycarbonate procedure and the cellulose ester filter procedure has been demonstrated for laboratory-generated aerosols of chrysotile asbestos.

10.3.3 Interlaboratory and intralaboratory analyses

Interlaboratory and intralaboratory analyses are required in order to monitor systematic errors that may develop among microscopists when using this method. These analyses should be designed to test both the overall method and the performance of individual microscopists. Repeating preparation of TEM grids from different sectors of a filter, followed by examination of the grids by a different microscopist, is a test for the reproducibility of the whole method. However, non-uniformity of the particulate deposit on the filter may lead to differences which are not related to the performance of the microscopists. Verified fibre counting (counting of asbestos structures on the same grid opening of a TEM grid by two or more operators, followed by resolution of any discrepancies) may be used both as a training aid and to determine the performance of different microscopists. The use of indexed TEM grids as described in 7.4.1 and 7.4.2 is recommended in order to facilitate relocation of specific grid openings.

10.4 Limit of detection

The limit of detection of the method can be varied by choice of the area of the collection filter, the volume of air sampled and the area of the specimen examined in the TEM. It is also a function of the background of asbestos structures on unused filters. A limit of detection shall be quoted for each sample analysis.

NOTE 12 In practice, the lowest limit of detection is frequently determined by the total suspended particulate concentration, since each particle on the filter must be separated from adjacent ones by a distance large enough for the particle to be identified without interference. Particulate loadings on sampling filters greater than 25 $\mu g/cm^2$ usually preclude preparation of TEM specimens by the direct methods. If the analysis is to be performed with an acceptable expenditure of time, the area of the specimen examined in the TEM for structures of all sizes is limited in most cases to between 10 and 20 grid openings. In typical ambient or building atmospheres, it has been found that an analytical sensitivity of 1 structure/l can be achieved. In some circumstances, where the atmosphere is exceptionally clean, this can be reduced to 0,1 structure/l or lower. For fibres and bundles longer than 5 µm, the reduced magnifications specified permit larger areas of the TEM specimens to be examined with an acceptable expenditure of time, resulting in proportionately lower limits of detection. If no structures are found in the analysis, the upper 95 % confidence limit can be quoted as the upper boundary of the concentration, corresponding to 2,99 times the analytical sensitivity if a Poisson distribution of structures on the filter is assumed. This 95 % confidence limit for 0 structures counted is taken as the detection limit. Since there is sometimes contamination of unused samples filters by asbestos structures, this should also be taken into account in the discussion of limits of detection.

11 Test report

The test report shall include at least the following information:

a) reference to this International Standard;

b) identification of the sample;

c) the date and time of sampling, and all necessary sampling data;

d) the date of the analysis;

e) the identity of the analyst;

f) any procedure used that is not specified in this International Standard or regarded as optional;

g) a complete listing of the structure counting data (the following data should be included: grid opening number, structure number, identification category, structure type, length and width of the structure in micrometres, and any comments concerning the structure);

h) a statement of the minimum acceptable identification category and the maximum identification category attempted (refer to tables D.1 and D.2);

i) a statement specifying which identification and structure categories have been used to calculate the concentration values;

j) separate concentration values for chrysotile and amphibole structures, expressed in number of asbestos structures per litre;

k) the 95 % confidence interval limits for the concentration values, expressed in number of asbestos structures per litre;

l) the analytical sensitivity, expressed in number of asbestos structures per litre;

m) the limit of detection, expressed in number of asbestos structures per litre;

n) compositional data for the principal varieties of amphibole, if present;

o) items g) to m) for asbestos fibres and bundles longer than 5 µm;

p) items g) to m) for PCM equivalent asbestos fibres and bundles.

An example of a suitable format for the structure counting data is shown in figures 6 and 7.

Sample analysis information (page 1)

Laboratory name	**Report number**	**Date**

Sample: 456 Queen Street
Ashby de la Zouch
Exterior sample 1991-09-09

Air volume:	2 150,0 litres
Area of collection filter:	385,0 mm^2
Level of analysis (chrysotile):	CD or CMQ
Level of analysis (amphibole):	ADQ
Magnification used for fibre counting:	× 20 500
Aspect ratio for fibre definition:	5/1
Mean dimension of grid openings:	95,4 µm
Initials of analyst:	JMW
Number of grid openings examined:	10
Analytical sensitivity:	1,968 structures/l
Number of primary asbestos structures:	13
Number of asbestos structures counted:	26
Number of asbestos structures > 5 µm :	7
Number of asbestos fibres and bundles > 5 µm :	10
Number of PCM equivalent asbestos structures:	3
Number of PCM equivalent asbestos fibres:	5

Figure 6 — Example of format for reporting sample and preparation data

Sample analysis information (pages 2 and following)

Laboratoriy name **Report number** **Date**

Sample: 456 Queen Street
Ashby de la Zouch
Exterior sample 1991-09-09

TEM asbestos structure count — Raw data

Grid	Grid opening	Number of structures		Identification[1)]	Structure type	Length	Width	Comments
		primary	total			µm	µm	
A	F4-4	1	1	CD	F	1,7	0,045	
		2	2	CMQ	B	2,6	0,09	
		3	3	ADQ	F	4,0	0,15	Crocidolite
	E3-6	4	4	CD	MC+0	3,5	1,3	
	E5-1	5		CD	MD43	7,5	5,0	
			5	CD	MB	7,7	0,30	
			6	CMQ	MF	5,6	0,045	
			7	CD	MB	5,1	0,30	
			8	CD	MF	1,7	0,045	
B	F4-1	6		CD	CD+0	6,5	3,0	
			9	CD	CB	3,5	0,15	
			10	CD	CF	3,5	0,045	
			11	CMQ	CR+0	2,6	1,9	
	G5-1	7		CD	CD31	6,1	3,2	
			12	CD	CB	5,6	0,3	
			13	CMQ	CF	4,0	0,045	
			14	CMQ	CB	3,2	0,090	
	E4-4	8	15	CD	B	1,5	0,23	
		9	16	AD	F	8,7	0,15	
C	G4-4	10		CMQ	CD42	25	5,6	
			17	CMQ	CB	15	0,15	
			18	CMQ	CF	9,4	0,045	
			19	ADQ	CF	3,6	0,30	Tremolite
			20	CM	CF	4,2	0,045	
	E4-4			No fibres				
	E5-6	11		ADQ	CD+3	9,4	2,5	
			21	ADQ	CF	7,1	0,30	Amosite
			22	ADQ	CF	6,2	0,10	Crocidolite
			23	CM	CB	5,1	0,2	
			24	CM	CR+0	3,3	1,8	
	F4-1	12	25	CMQ	MC10	3,7	2,1	
		13	26	CD	CC+0	7,4	0,5	

1) Identification codes listed in tables D.1 and D.2.

Figure 7 — Example of format for reporting structure counting data

Annex A
(normative)

Determination of operating conditions for plasma asher

A.1 General

During the preparation of TEM specimens from an MEC or cellulose nitrate filter, the spongy structure of the filter is collapsed into a thinner film of polymer by the action of a solvent. Some of the particles on the surface of the original filter become completely buried in the polymer, and the specimen preparation procedure incorporates a plasma etching step to oxidize the surface layer of the polymer. Particles buried by the filter collapsing step are then exposed so that they can become subsequently affixed to the evaporated carbon film without altering their position on the original filter. The amount of etching is critical, and individual ashers vary in performance. Therefore, the plasma asher (7.3.4) shall be calibrated to give a known amount of etching of the surface of the collapsed filter. This is carried out by adjusting the radio-frequency power output and the oxygen flow-rate, and measuring the time taken to completely oxidize an uncollapsed cellulose ester filter with 25 mm diameter of the same type and pore size as those used in the analysis.

A.2 Procedure

Place an unused cellulose ester filter, with 25 mm diameter, of the same type as that being used, in the centre of a glass microscope slide. Position the slide approximately in the centre of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 ml/min to 20 ml/min. Adjust the tuning of the system so that the intensity of the plasma in maximized. Measure the time required for complete oxidation of the filter. Determine operating parameters which result in complete oxydation of the filter in a period of approximately 15 min. For etching of collapsed filters, these operating parameters shall be used for a period of 8 min.

NOTE 13 Plasma oxidation at high radio-frequency powers will cause the filter to shrink and curl, followed by sudden violent ignition. At lower powers, the filter will remain in position and will slowly become thinner until it is nearly transparent. It is recommended that a radio-frequency power be used such that violent ignition does not occur. When multiple filters are etched, the rate of etching is reduced, and the system should be calibrated accordingly.

Annex B
(normative)

Calibration procedures

B.1 Calibration of the TEM

B.1.1 Calibration of TEM screen magnification

The electron microscope should be aligned according to the specifications of the manufacturer. Initially, and at regular intervals, calibrate the magnifications used for the analysis using a diffraction grating replica (7.3.11). Adjust the specimen height to the eucentric position before carrying out the calibration. Measure the distance on the fluorescent viewing screen occupied by a convenient number of repeat distances of the grating image, and calculate the magnification. Always repeat the calibration after any instrumental maintenance or change of operating conditions. The magnification of the image on the viewing screen is not the same as that obtained on photographic plates or film. The ratio between these is a constant value for the particular model of TEM.

B.1.2 Calibration of ED camera constant

Calibrate the camera constant of the TEM when used in ED mode. Use a specimen grid supporting a carbon film on which a thin film of gold has been evaporated or sputtered. Form an image of the gold film with the specimen adjusted to the eucentric position and select ED conditions. Adjust the objective lens current to optimize the pattern obtained, and measure the diameters of the innermost two rings either on the fluorescent viewing screen or on a recorded image. Calculate the radius-based camera constant, λL, for both the fluorescent screen and the photographic plate or film, using the following equation:

$$\lambda L = \frac{aD}{2{,}0\sqrt{h^2 + k^2 + l^2}}$$

where

- λ is the wavelength, in nanometres, of the incident electrons;
- L is the camera length, in millimetres;
- a is the unit cell dimension of gold, in nanometres (= 0,407 86 nm);
- D is the diameter, in millimetres, of the (hkl) diffraction ring.

Using gold as the calibration material, the radius-based camera constant is given by

$\lambda L = 0{,}117\ 74D$ mm·nm (smallest ring)

$\lambda L = 0{,}101\ 97D$ mm·nm (second ring)

B.2 Calibration of the EDXA system

Energy calibration of the EDXA system for a low energy and high energy peak shall be performed regularly. Calibration of the intensity scale of the EDXA system permits quantitative composition data, at an accuracy of about 10 % of the elemental concentration, to be obtained from EDXA spectra of reference silicate minerals involving the elements Na, Mg, Al, Si, K, Ca, Mn and Fe, and applicable certified reference materials. If quantitative determinations are required for minerals containing other elements, reference standards other than those referred to below will be required. Well-characterized mineral standards permit calibration of any TEM-EDXA combination which meets the instrumental specifications of 7.3.1 and 7.3.2, so that EDXA data from different instruments can be compared. Reference minerals are required for the calibration; the criteria for selection being that they should be silicate minerals with matrices as close as possible to those of the amphiboles or serpentine, and that small individual fragments of the minerals are homogeneous in composition within a few percent.

Determine the compositions of these standards by electron microprobe analysis or chemicals methods. Crush fragments of the same selected mineral standards and prepare filters by dispersal of the crushed material in water and immediate filtration of the suspensions. Prepare TEM specimens from these filters according to the procedures specified in clause 9. These TEM specimens can then be used to calibrate any TEM-EDXA system so that comparable compositional results can be obtained from different instruments.

NOTES

14 The microprobe analysis of the mineral standards are carried out by conventional techniques which can be found in annex J. The mineral is first embedded in a mount of poly(methyl methacrylate) or epoxy resin. The mount is then ground and polished to achieve a flat, polished surface of the mineral fragment. This surface is then analysed, using suitable reference standards, preferably oxide standards of the individual elements wherever these are available. It is necessary to take into account the water concentration in the minerals, which in the case of chrysotile amounts to 13 % by mass. This water content may vary due to losses in the vacuum system.

15 Aqueous suspensions of mineral standards should be filtered immediately after preparation, since alkali and alkali earth metals may be partially leached from minerals containing these elements.

Express the results of the electron microprobe analyses as atomic or mass percentage ratios relative to silicon. X-ray peak ratios of the same elements relative to silicon, obtained from the EDXA system, can then be used to calculate the relationship between peak area ratio and atomic or mass percentage ratio. The technique was described by Cliff and Lorimer (see annex J, reference [8]).

The X-rays generated in a thin specimen by an incident electron beam have a low probability of interacting with the specimen. Thus, mass absorption and fluorescence effects are negligible. In a silicate mineral specimen containing element i, the following equation can be used to perform quantitative analyses in the TEM:

$$\frac{C_i}{C_{Si}} = k_i \times \frac{A_i}{A_{Si}}$$

where

C_i is the concentration or atomic percentage of element i;

C_{Si} is the concentration or atomic percentage of silicon;

A_i is the elemental integrated peak area for element i;

A_{Si} is the elemental integrated peak area for silicon;

k_i is the k-ratio for element i relative to silicon.

For a particular instrumental configuration and a particular particle size, the value of k_i is constant.

To incorporate correction for the particle size effect on peak area ratios (see annex J, references [35] and [36], extend the Cliff and Lorimer technique by obtaining separate values of the constant k_i for different ranges of fibre diameter. It is recommended that 20 EDXA measurements be made for each range of fibre diameters. Suitable ranges of fibre diameter are:

$< 0,25$ µm; 0,25 µm to 0,5 µm; 0,5 µm to 1,0 µm; $> 1,0$ µm.

Insert the TEM grid into the transmission electron microscope, obtain an image at the calibrated higher magnification of about × 20 000, and adjust the specimen height to the eucentric point. If the X-ray detector is a side-entry variety, tilt the specimen towards the X-ray detector. Select an isolated fibre or particle less than 0,5 µm in width, and accumulate an EDXA spectrum using an electron probe of suitable diameter. When a well-defined spectrum has been obtained, perform a background subtraction and calculate the background-corrected peak areas for each element listed, using energy windows centred on the peaks. Calculate the ratio of the peak area for each specified element relative to the peak area for silicon. All background-subtracted peak areas used for calibration shall exceed 400 counts.

Repeat this procedure for 20 particles of each mineral standard. Reject analyses of any obviously foreign particles. Calculate the arithmetic mean concentration to peak area ratio, k_i (k-ratio), for each specified element of each mineral standard and for each of the fibre diameter ranges. Periodic routine checks shall be carried out to ensure that there has been no degradation of the detector performance. These k-ratios are used to calculate the elemental concentrations of unknown fibres, using the Cliff and Lorimer relationship.

Annex C
(normative)

Structure counting criteria

C.1 General

In addition to isolated fibres, other assemblages of particles and fibres frequently occur in air samples. Groupings of asbestos fibres and particles, referred to as "asbestos structures", are defined as fibre bundles, clusters and matrices. The numerical result of a TEM examination depends largely on whether the analyst assigns such an assemblage of fibres as a single entity, or as the estimated number of individual fibres which form the assemblage. It is therefore important that a logical system of counting criteria be defined, so that the interpretation of these complex structures is the same for all analysts, and so that the numerical result is meaningful. Imposition of specific structure-counting criteria generally requires that some interpretation, partially based on uncertain information on health effects, be made of each asbestos structure found. It is not the intention of this International Standard to make any interpretations based on health effects, and it is intended that a clear separation shall be made between recording of structure counting data, and later interpretation of those data. The system of coding specified in this International Standard permits a clear morphological description of the structures to be recorded in a concise manner suitable for later interpretation, if necessary, by a range of different criteria, without the necessity for re-examination of the specimens. In particular, the coding system is designed to permit the dimensions of each complex fibrous structure, and also whether these structures contain fibres longer than 5 µm, to be recorded. This approach permits later evaluations of the data to include considerations of particle respirability and comparisons with historical indices of asbestos exposure. Examples of the various types of morphological structure, and the manner in which these shall be recorded, are shown in figure C.1.

C.2 Structure definitions and treatment

Each fibrous structure that is a separate entity shall be designated as a primary structure. Each primary structure shall be designated as a fibre, bundle, cluster or matrix.

C.2.1 Fibre

Any particle with parallel or stepped sides, of minimum length 0,5 µm, and with an aspect ratio of 5/1 or greater, shall be defined as a fibre. For chrysotile asbestos, the single fibril shall be defined as a fibre. A fibre with stepped sides shall be assigned a width equal to the average of the minimum and maximum widths. This average shall be used as the width in determination of the aspect ratio.

C.2.2 Bundle

A grouping composed of apparently attached parallel fibres shall be defined as a bundle, with a width equal to an estimate of the mean bundle width, and a length equal to the maximum length of the structure. The overall aspect ratio of the bundle may have any value, provided that it contains individual constituent fibres with aspect ratios equal to or greater than 5/1. Bundles may exhibit diverging fibres at one or both ends.

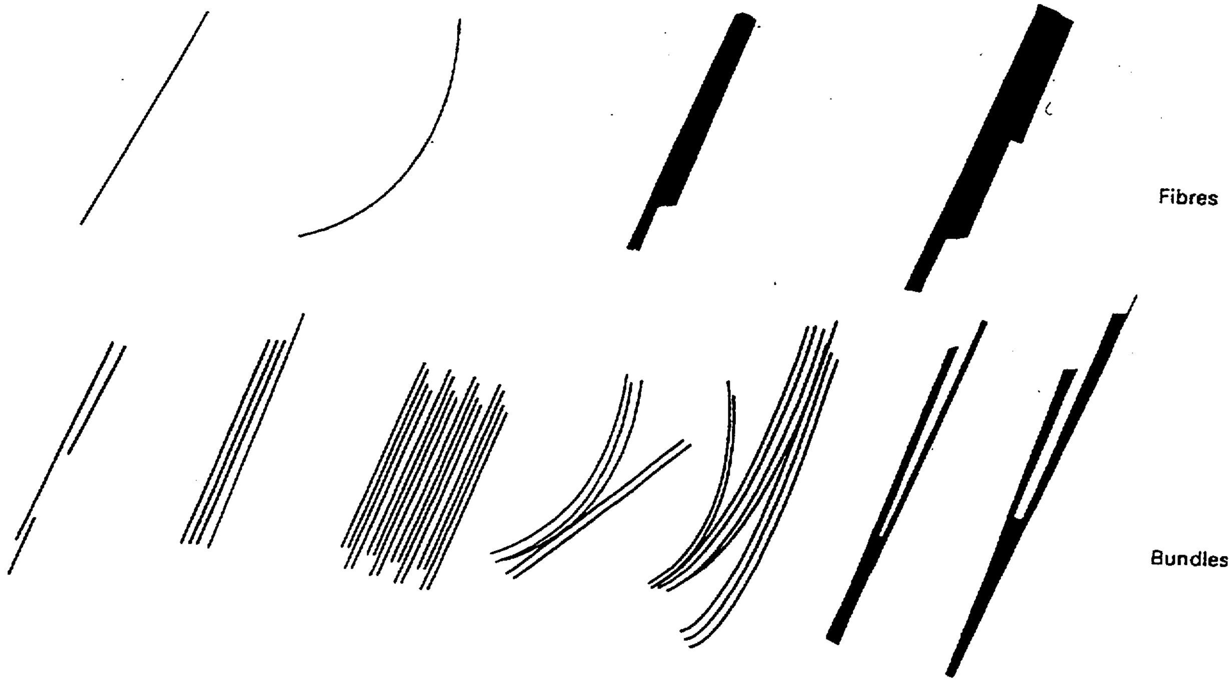
C.2.3 Cluster

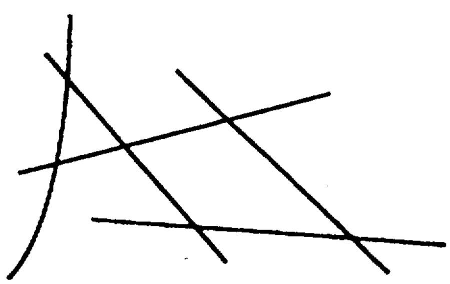
An aggregate of two or more randomly oriented fibres, with or without bundles, shall be defined as a cluster. Clusters occur as two varieties.

C.2.3.1 disperse cluster (type D): Disperse and open network, in which at least one of the individual fibres or bundles can be separately identified and its dimensions measured;

C.2.3.2 compact cluster (type C): Complex and tightly bound network, in which one or both ends of each individual fibre or bundle is (are) obscured, such that the dimensions of individual fibres and bundles cannot be unambiguously determined.

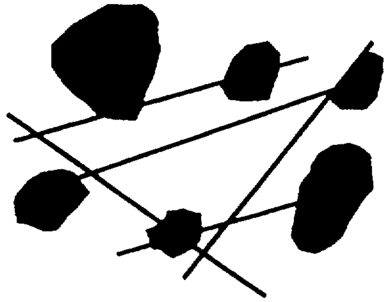
In practice, clusters can occur in which the characteristics of both types of cluster occur in the same structure. Where this occurs, the structure should be defined as a disperse cluster, and then a logical procedure should be followed by recording structure components according to the counting criteria. The procedure for treatment of clusters is illustrated by examples in figure C.2.



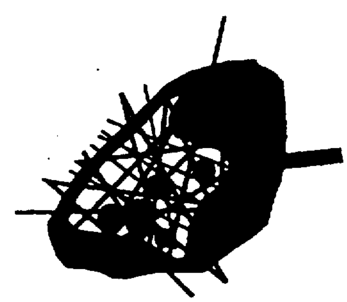


a) Disperse cluster (type D)

b) Compact cluster (type C)



c) Disperse matrix (type D)

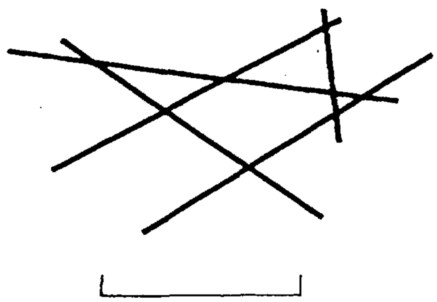


d) Compact matrix (type C)

Figure C.1 — Fundamental morphological structure types

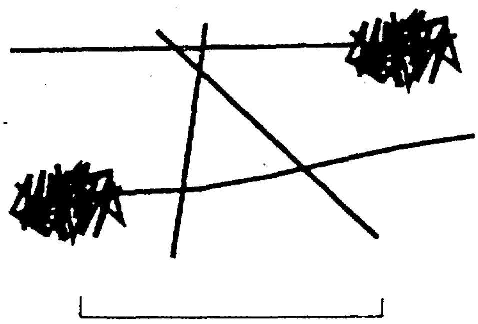
Count as 1 compact cluster containing more than 9 fibres (all fibres shorter than 5 µm)

Record as CC+0



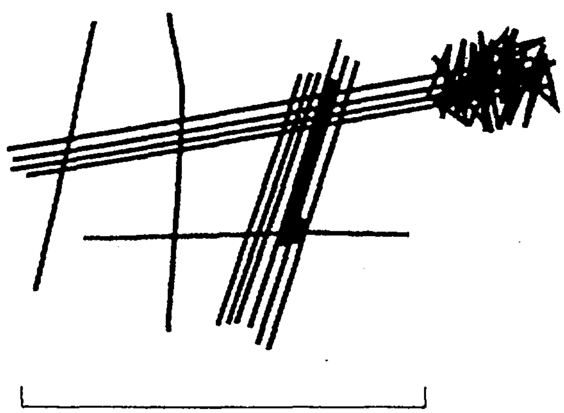
Count as 1 disperse cluster consisting of 5 fibres, 4 of which are longer than 5 µm

Record as CD54, followed by 5 fibres, each recorded as CF



Count as 1 disperse cluster consisting of 4 fibres, 2 of which are longer than 5 µm, and 2 cluster residuals, each containing more than 9 fibres

Record as CD+2, followed by 4 fibres, each recorded as CF, and 2 cluster residual, each recorded as CR+0



Count as 1 disperse cluster consisting of 3 fibres, 2 bundles, 1 of which is longer than 5 µm, and 1 cluster residual containing more than 9 fibres

Record as CD+1, followed by 3 fibres, each recorded as CF, 2 bundles, each recorded as CB, and 1 cluster residual recorded as CR+0

Figure C.2 — Examples of recording of complex asbestos clusters

C.2.4 Matrix

One or more fibres, or fibre bundles, may be attached to, or partially concealed by, a single particle or group of overlapping nonfibrous particles. This structure shall be defined as a matrix. The TEM image does not discriminate between particles which are attached to fibres, and those which have by chance overlapped in the TEM image. It is not known, therefore, whether such a structure is actually a complex particle, or whether it has arisen by a simple overlapping of particles and fibres on the filter.

Since a matrix structure may involve more than one fibre, it is important to define in detail how matrices shall be counted. Matrices exhibit different characteristics, and two types can be defined.

C.2.4.1 disperse matrix (type D): Structure consisting of a particle or linked group of particles, with overlapping or attached fibres or bundles in which at least one of the individual fibres or bundles can be separately identified and its dimensions measured.

C.2.4.2 compact matrix (type C): Structure consisting of a particle or linked group of particles, in which fibres or bundles can be seen either within the structure or projecting from it, such that the dimensions of individual fibres and bundles cannot be unambiguously determined.

In practice, matrices can occur in which the characteristics of both types of matrix occur in the same structure. Where this occurs, the structure should be assigned as a disperse matrix, and then a logical procedure should be followed by recording structure components according to the counting criteria. Examples of the procedure which shall be followed are shown in figure C.3.

C.2.5 Asbestos structure larger than 5 µm

Any fibre, bundle, cluster or matrix for which the largest dimension exceeds 5 µm. Asbestos structures larger than 5 µm do not necessarily contain asbestos fibres or bundles longer than 5 µm.

C.2.6 Asbestos fibre or bundle longer than 5 µm

An asbestos fibre of any width, or bundle of such fibres, which has a length exceeding 5 µm.

C.2.7 PCM equivalent structure

Any fibre, bundle, cluster or matrix with an aspect ratio of 3/1 or greater, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm. PCM equivalent structures do not necessarily contain fibres or bundles longer than 5 µm, or PCM equivalent fibres.

C.2.8 PCM equivalent fibre

Any particle with parallel or stepped sides, with an aspect ratio of 3/1 or greater, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm. For chrysotile, PCM equivalent fibres will always be bundles.

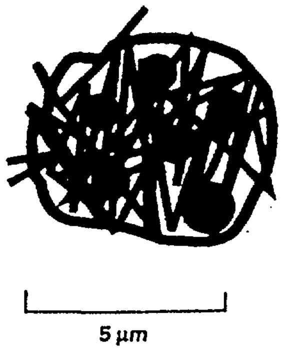
C.3 Other structure counting criteria

C.3.1 Structures which intersect grid bars

A structure which intersects a grid bar shall only be counted on two sides of the grid opening, as illustrated in figure C.4. Record the dimensions of the structure such that the obscured portions of components are taken to be equivalent to the unobscured portions, as shown by the broken lines in figure C.4. For example, the length of a fibre intersecting a grid bar is taken to be twice the unobscured length. Structures intersecting either of the other two sides shall not be included in the count.

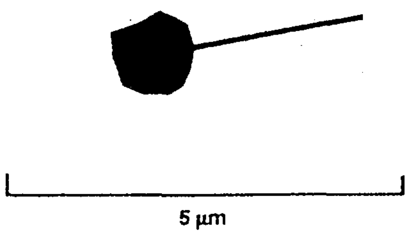
C.3.2 Fibres which extend outside the field of view

During scanning of a grid opening, count fibres which extend outside the field of view systematically, so as to avoid double-counting. In general, a rule should be established so that fibres extending outside the field of view in only two quadrants are counted. The procedure is illustrated by figure C.5. Measure the length of each of these fibre by moving the specimen to locate the other end of the fibre, and then return to the original field of view before continuing to scan the specimen. Fibres without terminations within the field of view shall not be counted.



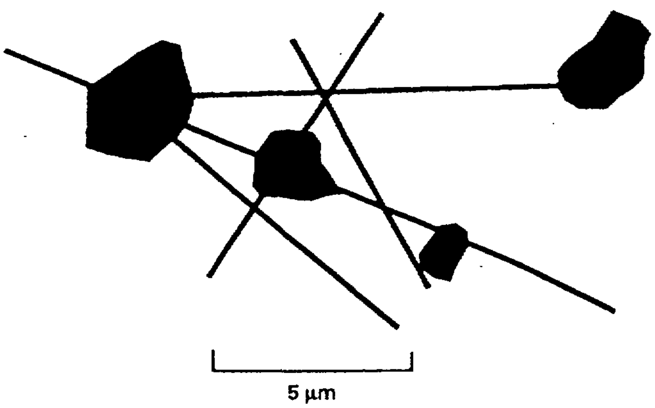
Count as 1 compact matrix, with all fibres shorter than 5 µm

Record as MC+0



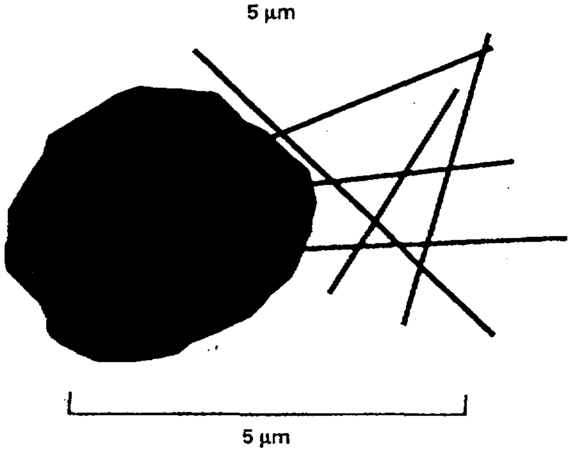
Count as 1 disperse matrix consisting of 1 fibre shorter than 5 µm

Record as MD10, followed by 1 fibre recorded as MF



Count as 1 disperse matrix consisting of 5 fibres, all longer than 5 µm

Record as MD55, followed by 5 fibres, each recorded as MF



Count as 1 disperse matrix, consisting if 3 fibres, 1 of which is longer than 5 µm, and 1 matrix residual containing 3 fibres

Record as MD61, followed by 3 fibres, each recorded as MF, and 1 matrix residual recorded as MR30

Figure C.3 — Examples of recording of complex asbestos matrices

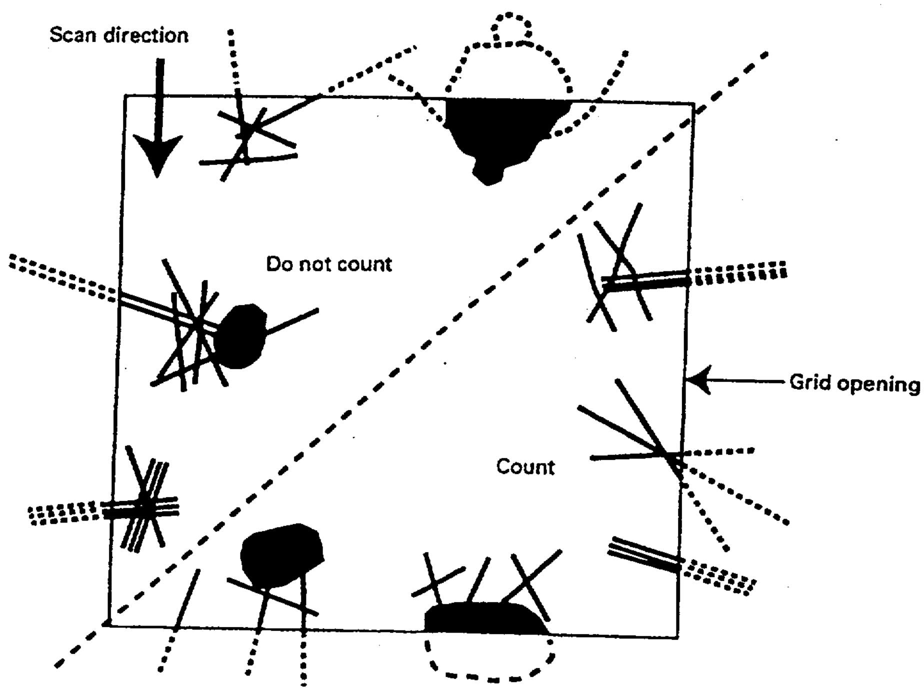


Figure C.4 — Example of counting of structures which intersect grid bars

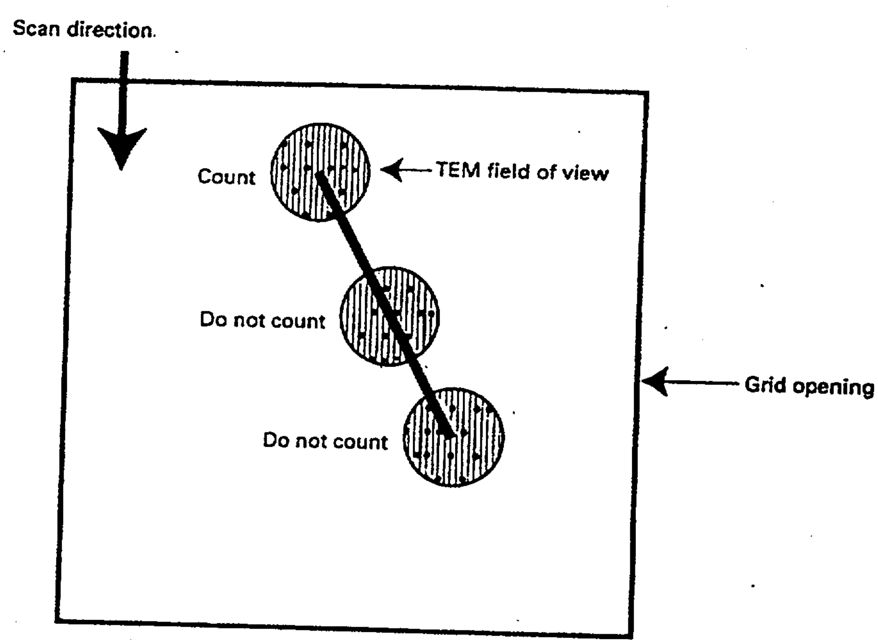


Figure C.5 — Example of counting of fibres which extend outside the field of view

C.4 Procedure for data recording

C.4.1 General

The morphological codes specified are designed to facilitate computer data processing, and to allow recording of a complete representation of the important features of each asbestos structure. The procedure requires that the microscopist classify each primary fibrous structure into one of the four fundamental categories: fibres, bundles, clusters and matrices.

C.4.2 Fibres

On the structure counting form, a fibre as defined in C.2.1 shall be recorded by the designation "F". If the fibre is a separately-counted part of a cluster or matrix, the fibre shall be recorded by the designation "CF", or "MF", depending on whether it is a component of a cluster or matrix.

C.4.3 Bundles

On the structure counting form, a bundle as defined in C.2.2 shall be recorded by the designation "B". If the bundle is a separately-counted part of a cluster or matrix, the bundle shall be recorded by the designation "CB", or "MB", depending on whether it is a component of a cluster or matrix.

C.4.4 Disperse clusters (type D)

On the structure counting form, an isolated cluster of type D as defined in C.2.3 shall be recorded by the designation "CD", followed by a two-digit number. The first digit represents the analyst's estimate of the total number of fibres and bundles comprising the structure. The digit shall be from 1 to 9, or designated as "+" if there are estimated to be more than 9 component fibres or bundles. The second digit shall represent, in the same manner, the total number of fibres and bundles longer than 5 µm contained in the structure. The overall dimensions of the cluster, in two perpendicular directions representing the maximum dimensions, shall be recorded. In order of decreasing length, up to 5 component fibres or bundles shall be separately recorded, using the codes "CF" (cluster fibre) and "CB" (cluster bundle). If, after accounting for prominent component fibres and bundles, a group of clustered fibres remains, this shall be recorded by the designation "CR" (cluster residual). If the remaining clustered fibres are present as more than one localized group, it may be necessary to record more than one cluster residual. Do not record more than 5 cluster residuals for any cluster. A cluster residual shall be measured and assigned a two-digit number, derived in the same manner as specified for the overall cluster. Optionally, if the number of component fibres and bundles in either the original cluster or the cluster residual is outside the range 1 – 9, additional information concerning the number of component fibres and bundles may be noted in the "comments" column.

C.4.5 Compact clusters (type C)

On the structure counting form, an isolated cluster of type C as defined in C.2.3 shall be recorded by the designation "CC", followed by a two-digit number. The two-digit number describing the numbers of component fibres and bundles shall be assigned in the same manner as for clusters of type D. The overall dimensions of the cluster in two perpendicular directions shall be recorded in the same manner as for clusters of type D. By definition, the constitutent fibres and bundles of compact clusters cannot be separately measured; therefore, no separate tabulation of component fibres or bundles can be made.

C.4.6 Disperse matrices (type D)

On the structure counting form, an isolated matrix of type D as defined in C.2.4 shall be recorded by the designation "MD", followed by a two-digit number. The two-digit number shall be assigned in the same manner as for clusters of type D. The overall dimensions of the matrix in two perpendicular directions shall be recorded in the same manner as for clusters of type D. In order of decreasing length, up to 5 component fibres or bundles shall be separately recorded, using the codes "MF" (matrix fibre) and "MB" (matrix bundle). If after accounting for prominent component fibres and bundles, matrix material containing asbestos fibres remains, this shall be recorded by the designation "MR" (matrix residual). If the remaining matrix fibres are present as more than one localized group, it may be necessary to record more than one matrix residual. Do not record more than 5 matrix residuals for any matrix. A matrix residual shall be measured and assigned a two-digit number, derived in the same manner as specified for the overall matrix. Optionally, if the number of component fibres or bundles in either the original matrix or the matrix residual is outside the range 1 – 9, additional information concerning the number of component fibres and bundles may be noted in the "comments" column.

C.4.7 Compact matrices (type C)

On the structure counting form, an isolated matrix of type C as defined in C.2.4 shall be recorded by the designation "MC", followed by a two-digit number. The two-digit number shall be assigned in the same manner as for clusters of type D. The overall dimensions of the matrix in two perpendicular directions shall be recorded in the same manner as for clusters of type D. By definition, the constitutent fibres and bundles of compact matrices cannot be separately measured; therefore, no separate tabulation of component fibres or bundles can be made.

C.4.8 Procedure for recording of partially obscured fibres and bundles

The proportion of the length of a fibre or bundle that is obscured by other particulates shall be used as the basis for determining whether a fibre or bundle is to be recorded as a separate component or is to be considered as a part of a matrix of type C or part of a matrix residual. If the obscured length could not possibly be more than one-third of the total length, the fibre or bundle shall be considered a prominent feature to be separately recorded. The assigned length for each such partially obscured fibre or bundle shall be equal to the visible length plus the maximum possible contribution from the obscured portion. Fibres or bundles which appear to cross the matrix, and for which both ends can be located approximately, shall be included in the maximum of 5 and recorded according to the counting criteria as separate fibres or bundles. If the obscured length could be more than one third of the total length, the fibre or bundle shall be considered as a part of a compact matrix of type C or part of a matrix residual.

C.5 Special considerations for counting of PCM equivalent structures

Use 3/1 as the minimum aspect ratio for counting of PCM equivalent structures. This aspect ratio definition is required in order to achieve comparability of the results for this size range of structure with historical optical measurements, but use of this aspect ratio definition does not significantly affect the ability to interpret the whole fibre size distribution in terms of a minimum 5/1 aspect ratio. Some applications may require that a count be made of PCM equivalent structures only. The coding system permits discrimination between PCM equivalent structures that contain fibres and bundles longer than 5 µm and those that do not.

NOTE 16 In general, clusters and matrices will yield fewer components as the minimum dimensions specified for countable fibres are increased. Thus, it may be found that a particular structure yields a higher number of component fibres and bundles in a count for all fibre sizes than it does at a reduced magnification when only fibres and bundles longer than 5 µm are being counted. However, the requirement that component fibres and bundles be recorded in decreasing length order ensures that the data are consistent for a particular structure, regardless of the size category of fibres being counted and the magnification in use.

Annex D
(normative)

Fibre identification procedure

D.1 General

The criteria used for identification of asbestos fibres may be selected depending on the intended use of the measurements. In some circumstances, there can be a requirement that fibres shall be unequivocally identified as a specific mineral species. In other circumstances, there can be sufficient knowledge about the sample, so that rigorous identification of each fibre need not be carried out. The time required to perform the analysis, and therefore the cost of analysis, can vary widely depending on the identification criteria considered which are to be sufficiently definitive. The combination of criteria considered definitive for identification of fibres in a particular analysis shall be specified before the analysis is made, and this combination of criteria shall be referred to as the "level" of analysis. Various factors related to instrumental limitations and the character of the sample may prevent satisfaction of all of the specified fibre identification criteria for a particular fibre. Therefore, a record shall be made of the identification criteria which were satisfied for each suspected asbestos fibre included in the analysis. For example, if both ED and EDXA were specified to be attempted for definitive identification of each fibre, fibres with chrysotile morphology which, for some reason, do not give an ED pattern but which do yield an EDXA spectrum corresponding to chrysotile, are categorized in a way which conveys the level of confidence to be placed in the identification.

D.2 ED and EDXA techniques

D.2.1 General

Initially, fibres are classified into two categories on the basis of morphology: those fibres with tubular morphology, and those fibres without tubular morphology. Further analysis of each fibre is conducted using ED and EDXA methods. The following procedures should be used when fibres are examined by ED and EDXA.

The crystal structures of some mineral fibres, such as chrysotile, are easily damaged by the high current densities required for EDXA examination. Therefore, investigation of these sensitive fibres by ED should be completed before attempts are made to obtain EDXA spectra from the fibres. When more stable fibres, such as the amphiboles, are examined, EDXA and ED may be used in either order.

D.2.2 ED techniques

The ED technique can be either qualitative or quantitative. Qualitative ED consists of visual examination, without detailed measurement, of the general characteristics of the ED pattern obtained on the TEM viewing screen from a randomly oriented fibre. ED patterns obtained from fibres with cylindrical symmetry, such as chrysotile, do not change when the fibres are tilted about their axes, and patterns from randomly oriented fibres of these minerals can be interpreted quantitatively. For fibres which do not have cylindrical symmetry, only those ED patterns obtained when the fibre is oriented with a principal crystallographic axis closely parallel with the incident electron beam direction can be interpreted quantitatively. This type of ED pattern shall be referred to as a "zone-axis ED pattern". In order to interpret a zone-axis ED pattern quantitatively, it shall be recorded photographically and its consistency with known mineral structures shall be checked. A computer program may be used to compare measurements of the zone-axis ED pattern with corresponding data calculated from known mineral structures. The zone-axis ED pattern obtained by examination of a fibre in a particular orientation can be insufficiently specific to permit unequivocal identification of the mineral fibre, but is is often possible to tilt the fibre to another angle and to record a different ED pattern corresponding to another zone-axis. The angle between the two zone-axes can also be checked for consistency with the structure of a suspected mineral.

For visual examination of the ED pattern, the camera length of the TEM should be set to a low value of approximately 250 mm and the ED pattern should then be viewed through the binoculars. This procedure minimizes the possible degradation of the fibre by the electron irradiation. However, the pattern is distorted by the tilt angle of the viewing screen. A camera length of at least 2 m should be used when the ED pattern is recorded, if accurate measurement of the pattern is to be possible. It is necessary that, when obtaining an ED pattern to be evaluated visually or to be recorded, the sample height shall be properly adjusted to the eucentric point and the image shall be focussed in the plane of the selected area aperture. If this is not done, there may be some components of the ED pattern which do not originate from the selected area. In general, it will be necessary to use the smallest available ED aperture.

For accurate measurements of the ED pattern, an internal calibration standard shall be used. A thin coating of gold, or another suitable calibration material, shall be applied to the underside of the TEM specimen. This coating may be applied either by vacuum evaporation or, more conveniently, by sputtering. The polycrystalline gold film yields diffraction rings on every ED pattern and these rings provide the required calibration information.

To form an ED pattern, move the image of the fibre to the centre of the viewing screen, adjust the height of the specimen to the eucentric position, and insert a suitable selected area aperture into the electron beam so that the fibre, or a portion of it, occupies a large proportion of the illuminated area. The size of the aperture and the portion of the fibre shall be such that particles other than the one to be examined are excluded from the selected area. Observe the ED pattern through the binoculars. During the observation, the objective lens current should be adjusted to the point where the most complete ED pattern is obtained. If an incomplete ED pattern is still obtained, move the particle around within the selected area to attempt to optimize the ED pattern, or to eliminate possible interferences from neighbouring particles.

If a zone-axis ED analysis is to be attempted on the fibre, the sample shall be mounted in the appropriate holder. The most convenient holder allows complete rotation of the specimen grid and tilting of the grid about a single axis. Rotate the sample until the fibre image indicates that the fibre is oriented with its length coincident with the tilt axis of the goniometer, and adjust the sample height until the fibre is at the eucentric position. Tilt the fibre until an ED appears which is a symmetrical, two dimensional array of spots. The recognition of zone-axis alignment conditions requires some experience on the part of the operator. During tilting of the fibre to obtain zone-axis conditions, the manner in which the intensities of the spots vary should be observed. If weak reflections occur at some points on a matrix of strong reflections, the possibility of twinning or multiple diffraction exists, and some caution should be exercised in the selection of diffraction spots for measurement and interpretation. A full discussion of electron diffraction and multiple diffraction can be found in the references by J.A. Gard [11] P.B. Hirsch *et al* [14] and H.R. Wenck [42] included in annex J. Not all zone-axis patterns which can be obtained are definitive. Only those which have closely spaced reflections corresponding to low indices in at least one direction should be recorded. Patterns in which all d-spacings are less than about 0,3 nm are not definitive. A useful guideline is that the lowest angle reflections should be within the radius of the first gold diffraction ring (111), and that patterns with smaller distances between reflections are usually the most definitive.

Five spots, closest to the centre spot, along two intersecting lines of the zone-axis pattern shall be selected for measurement, as shown in figure D.1. The distances of these spots from the centre spot and the four angles shown provide the required data for analysis. Since the centre spot is usually very overexposed, it does not provide a well-defined origin for these measurements. The required distances shall therefore be obtained by measuring between pairs of spots symmetrically disposed about the centre spot preferably separated by several repeat distances. The distances shall be measured with a precision of better than 0,3 mm, and the angles to a precision of better than 2,5°. The diameter of the first or second ring of the calibration pattern (111 and 200) shall also be measured with a precision of better than 0,3 mm.

Using gold as the calibration material, the radius-based camera constant is given by

$$\lambda L = 0{,}117\ 74D \text{ mm·nm (first ring)}$$

$$\lambda L = 0{,}101\ 97D \text{ mm·nm (second ring)}$$

D.2.3 EDXA measurements

Interpretation of the EDXA spectrum may be either qualitative or quantitative. For qualitative interpretation of a spectrum, the X-ray peaks originating from the elements in the fibre are recorded. For quantitative interpretation, the net peak areas, after background subtraction, are obtained for the X-ray peaks originating from the elements in the fibre. This method provides quantitative interpretation for those minerals which contain silicon.

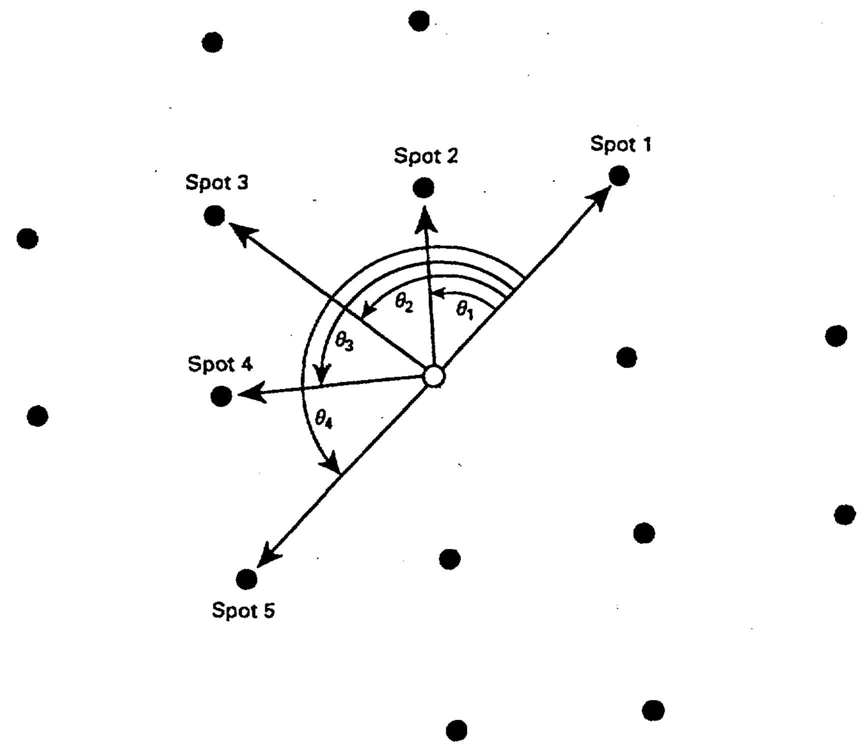


Figure D.1 — Example of measurement of zone-axis SAED patterns

To obtain an EDXA spectrum, move the image of the fibre to the centre of the screen and remove the objective aperture. Select an appropriate electron beam diameter and deflect the beam so that it impinges on the fibre. Depending on the instrumentation, it may be necessary to tilt the specimen towards the X-ray detector and, in some instruments, to use the Scanning Transmission Electron Microscopy (STEM) mode of operation.

The time for acquisition of a suitable spectrum varies with the fibre diameter, and also with instrumental factors. For quantitative interpretation, spectra should have a statistically valid number of counts in each peak. Analyses of small diameter fibres which contain sodium are the most critical, since it is in the low energy range that the X-ray detector is least sensitive. Consequently, it is necessary to acquire a spectrum for a period that is sufficiently long for the sodium to be detected in such fibres. It has been found that satisfactory quantitative an analyses can be obtained if acquisition is continued until the background subtracted silicon $K\alpha$ peak integral exceeds 10 000 counts. The spectrum should then be manipulated to subtract the background and to obtain the net areas of the elemental peaks.

After quantitative EDXA classification of some fibres by computer analysis of the net peak areas, it may be possible to classify further fibres in the same sample on the basis of comparison of spectra at the intrument. Frequently, visual comparisons can be made after somewhat shorter acquisition times.

D.3 Interpretation of fibre analysis data

D.3.1 Chrysotile

The morphological structure of chrysotile is characteristic, and with experience, can be recognized readily. However, a few holder minerals have a similar appearance, and morphological observation by itself is inadequate for most samples. The ED pattern obtained from chrysotile is quite specific for this mineral if the specified characteristics of the pattern correspond to those from reference chrysotile. However, depending on the past history of the fibre, and on a number of other factors, the crystal structure of a particular fibre may be damaged, and it may not yield and ED pattern. In this case, the EDXA spectrum may be the only data available to supplement the morphological observations.

D.3.2 Amphiboles

Since the fibre identification procedure for asbestos fibres other than chrysotile can be involved and time-consuming, computer programmes, such as that developed by B.L. Rhoades (see annex J, reference

[32]), are recommended for interpretation of zone-axis ED patterns. The published literature contains composition and crystallographic data for all of the fibrous minerals likely to be encountered in TEM analysis of air samples, and the compositional and structural data from the unknown fibre should be compared with the published data. Demonstration that the measurements are consistent with the data for a particular test mineral does not uniquely identify the unknown, since the possibility exists that data from other minerals may also be consistent. It is, however, unlikely that a mineral of another structural class could yield data consistent with that from an amphibole fibre identified by quantitative EDXA and two zone-axis ED patterns.

Suspected amphibole fibres should be classified initially on the basis of chemical composition. Either qualitative or quantitative EDXA information may be used as the basis for this classification. From the published data on mineral compositions, a list of minerals which are consistent in composition with that measured for the unknown fibre should be compiled. To proceed further, it is necessary to obtain the first zone-axis ED pattern, according to D.2.2.

It is possible to specify a particular zone-axis pattern for identification of amphibole, since a few patterns are often considered to be characteristic. Unfortunately, for a fibre with random orientation on a TEM grid, no specimen holder and goniometer currently available will permit convenient and rapid location of two preselected zone-axes. The most practical approach has been adopted, which is to accept those low index patterns which are easily obtained, and then to test their consistency with the structures of the minerals already preselected on the basis of the EDXA data. Even the structures of non-amphibole minerals in this preselected list shall be tested against the zone-axis data obtained for the unknown fibre, since non-amphibole minerals in some orientations may yield similar patterns consistent with amphibole structures.

The zone-axis ED interpretation shall include all minerals previously selected from the mineral data file as being chemically compatible with the EDXA data. This procedure will usually shorten the list of minerals for which solutions have been found. A second set of zone-axis data from another pattern obtained on the same fibre can then be processed, either as further confirmation of the identification, or to attempt elimination of an ambiguity. In addition, the angle measured between the orientations of the two zone-axes can be checked for consistency with the structures of the minerals. Caution should be exercised in rationalizing the inter-zone-axis angle, since if the fibre contains $\vec{c}$-axis twinning, the two zone-axis ED patterns may originate from the separate twin crystals. In practice, the full identification procedure will normally be applied to very few fibres, unless precise identification of all fibres is required for a particular reason.

D.4 Fibre classification categories

It is not always possible to proceed to a definitive identification of a fibre; this may be due to instrumental limitations or to the actual nature of the fibre. In many analyses, a definitive identification of each fibre may not actually be necessary if there is other knowledge available about the sample, or if the concentration is below a level of interest. The analytical procedure shall therefore take into account both instrumental limitations and varied analytical requirements. Accordingly, a system for fibre classification is used to permit accurate recording of data. The classifications are shown in tables D.1 and D.2, and are directed towards identification of chrysotile and amphibole respectively. Fibres shall be reported in these categories.

The general principle to be followed in this analytical procedure is first to define the most specific fibre classification which is to be attempted, or the "level" of analysis to be conducted. Then, for each fibre examined, record the classification which is actually achieved. Depending on the intended use of the results, criteria for acceptance of fibres as "identified" can then be established at any time after completion of the analysis.

In an unknown sample, chrysotile will be regarded as confirmed only if a recorded, calibrated ED pattern from one fibre in the CD categories is obtained, or if measurements of the ED pattern are recorded at the instrument. Amphibole will be regarded as confirmed only by obtaining recorded data which indicates exclusively the presence of amphiboles for fibres classified in the AZQ, AZZ or AZZQ categories.

Table D.1 — Classification of fibres with tubular morphology

Category	Description
TM	Tubular Morphology, not sufficiently characteristic for classification as chrysotile
CM	Characteristic Chrysotile Morphology
CD	Chrysotile SAED pattern
CQ	Chrysotile composition by Quantitative EDXA
CMQ	Chrysotile Morphology and composition by Quantitative EDXA
CDQ	Chrysotile SAED pattern and composition by Quantitative EDXA
NAM	Non-Asbestos Mineral

Table D.2 — Classification of fibres without tubular morphology

Category	Description
UF	Unidentified Fibre
AD	Amphibole by random orientation SAED (shows layer pattern of 0,53 nm spacing)
AX	Amphibole by qualitative EDXA. Spectrum has elemental components consistent with amphibole
ADX	Amphibole by random orientation SAED and qualitative EDXA
AQ	Amphibole by Quantitative EDXA
AZ	Amphibole by one Zone-axis SAED pattern
ADQ	Amphibole by random orientation SAED and Quantitative EDXA
AZQ	Amphibole by one Zone-axis SAED pattern and Quantitative EDXA
AZZ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle
AZZQ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle, and Quantitative EDXA
NAM	Non-Asbestos Mineral

D.4.1 Procedure for classification of fibres with tubular morphology suspected to be chrysotile

Occasionally, fibres are encountered which have tubular morphology similar to that of chrysotile, but which cannot be characterized further either by ED or EDXA. They may be non-crystalline, in which case ED techniques are not useful, or they may be in a position on the grid which does not permit an EDXA spectrum to be obtained. Alternatively, the fibre may be of organic origin, but the morphology and composition may not be sufficiently definitive enough to be disregarded. Accordingly, there is a requirement to record each fibre, and to specify how confidently each fibre can be identified. Classification of fibres will meet with various degrees of success. Figure D.2 shows the classification procedure to be used for fibres which display any tubular morphology. The chart is self explanatory, and every fibre is either rejected as a non-asbestos mineral (NAM), or classified in some way which by some later criterion could still contribute to the chrysotile fibre count.

Morphology is the first consideration, and if this is not similar to that usually seen in chrysotile standard samples, designate the initial classification as TM. Regardless of the doubtful morphology, examine the fibre by ED and EDXA methods according to figure D.2. Where the morphology is more definitive, it may be possible to classify the fibre as having chrysotile morphology (CM).

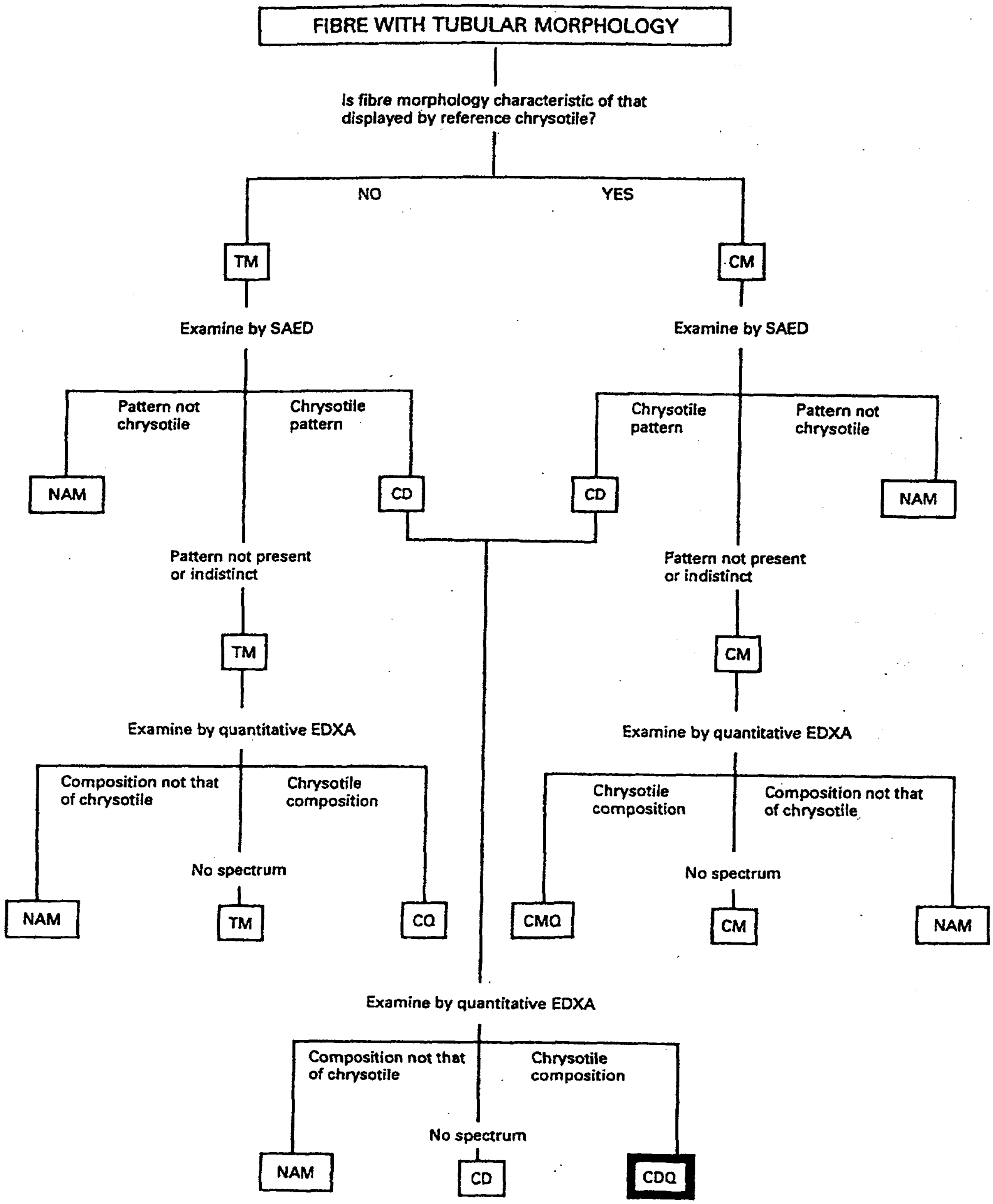


Figure D.2 — Classification chart for fibre with tubular morphology

For classification as CM, the morphological characteristics required are the following:

a) the individual fibrils should have high aspect ratios exceeding 5/1, and be about 30 nm to 40 nm in diameter;

b) the electron scattering power of the fibre at 60 kV to 100 kV accelerating potential should be sufficiently low for the internal structure to be visible;

c) there should be some evidence of an internal structure suggesting a tubular appearance similar to that shown by reference UICC chrysotile, which may degrade in the electron beam.

Examine every fibre having these morphological characteristics by the ED technique, and classify as chrysotile by ED (CD) only those which give diffraction patterns with the precise characteristics shown in figure D.3. The relevant features in this pattern for identification of chrysotile are as follows:

a) the (002) reflections should be examined to determine that they correspond closely to a spacing of 0,73 nm;

b) the layer line repeat distance should correspond to 0,53 nm;

c) there should be "streaking" of the (110) and (130) reflections.

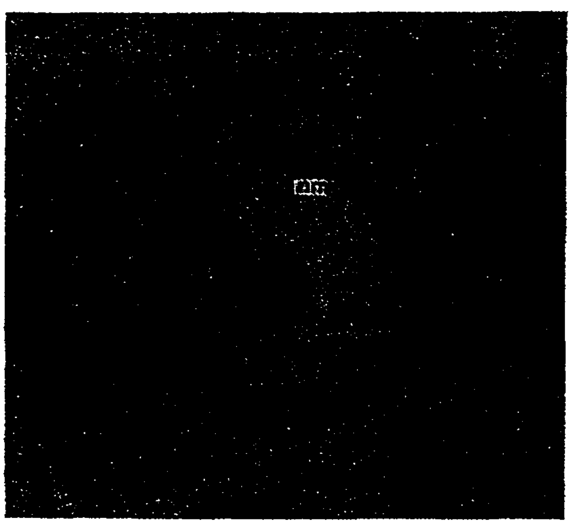


Figure D.3 — Chrysotile SAED pattern

Using the millimetre calibrations on the TEM viewing screen, these observations can readily be made at the instrument. If documentary proof of fibre identification is required, record a TEM micrograph of at least one representative fibre, and record its ED pattern on a separate film or plate. This film or plate shall also carry calibration rings from a known polycrystalline substance such as gold. This calibrated pattern is the only documentary proof that the particular fibre is chrysotile, and not some other tubular or scrolled species such as halloysite, palygorskite, talc or vermiculite. The proportion of fibres which can be successfully identified as chrysotile by ED is variable, and to some extent dependent on both the instrument and the procedures of the operator. The fibres that fail to yield an identifiable ED pattern will remain in the TM or CM categories unless they are examined by EDXA.

In the EDXA analysis of chrysotile there are only two elements which are relevant. For fibre classification, the EDXA analysis shall be quantitative. If the spectrum displays prominent peaks from magnesium and silicon, with their areas in the appropriate ratio, and with only minor peaks from other elements, classify the fibre as chrysotile by quantitative EDXA, in the categories CQ, CMQ, or CDQ, as appropriate.

D.4.2 Procedure for classification of fibres without tubular morphology, suspected to be amphibole

Every particle without tubular morphology and which is not obviously of biological origin, with an aspect ratio of 5/1 or greater, and having parallel or stepped sides, shall be considered as a suspected amphibole fibre. Further examination of the fibre by ED and EDXA techniques will meet with a variable degree of success, depending on the nature of the fibre and on a number of instrumental limitations. It will not be possible to identify every fibre completely, even if time and cost are of no concern. Moreover, confirmation of the presence of amphibole can be achieved only by quantitative interpretation of zone-axis ED patterns, a very time-consuming procedure. Accordingly, for routine samples from unknown sources, this analytical procedure limits the requirement for zone-axis ED work to a minimum of one fibre representative of each compositional class reported. In some samples, it may be necessary to identify more fibres by the zone-axis technique. When analysing samples from well-characterized sources, the cost of identification by zone-axis methods may not be justified.

The 0,53 nm layer spacing of the random orientation ED pattern is not by itself diagnostic for amphibole. However, the presence of $\vec{c}$-axis twinning in many fibres leads to contributions to the layers in the patterns by several individual parallel crystals of different axial orientations. This apparently random positioning of the spots along the layer lines, if also associated with a high fibre aspect ratio, is a characteristic of amphibole asbestos, and thus has some limited diagnostic value. If a pattern of this type is not obtained, the identity of the fibre is still ambiguous, since the absence of a recognizable pattern may be a consequence of an unsuitable orientation relative to the electron beam, or the fibre may be some other mineral species.

Figure D.4 shows the fibre classification chart to be used for suspected amphibole fibres. This chart shows all the classification paths possible in analysis of a suspected amphibole fibre, when examined systematically by ED and EDXA. Two routes are possible, depending on whether an attempt to obtain an EDXA spectrum or a random orientation ED pattern is made first. The normal procedure for analysis of a sample of unknown origin will be to examine the fibre by random orientation ED, qualitative EDXA, quantitative EDXA, and zone-axis ED, in this sequence. The final fibre classification assigned will be defined either by successful analysis at the maximum required level, or by the instrumental limitations. Any instrumental limitations which affect the quality of the results sh be noted. Record the maximum classificatio achieved for each fibre on the counting sheet in the appropriate column. The various classification categories can then be combined later in any desired way for calculation of the fibre concentration. The complete record of the results obtained when attempting to identify each fibre can also be used to re-assess the data if necessary.

In the unknown sample, zone-axis analysis will be required if the presence of amphibole is to be unequivocally confirmed. For this level of analysis, attempt to raise the classification of every suspected amphibole fibre to the ADQ category by inspection of the random orientation ED pattern and the EDXA spectrum. In addition, examine at least one fibre from each type of suspected amphibole found by zone-axis methods to confirm their identification. In most cases, because information exists about possible sources of asbestos in close proximity to the air sampling location, some degree of ambiguity of identification can be accepted. Lower levels of analysis can therefore be accepted for these situations.

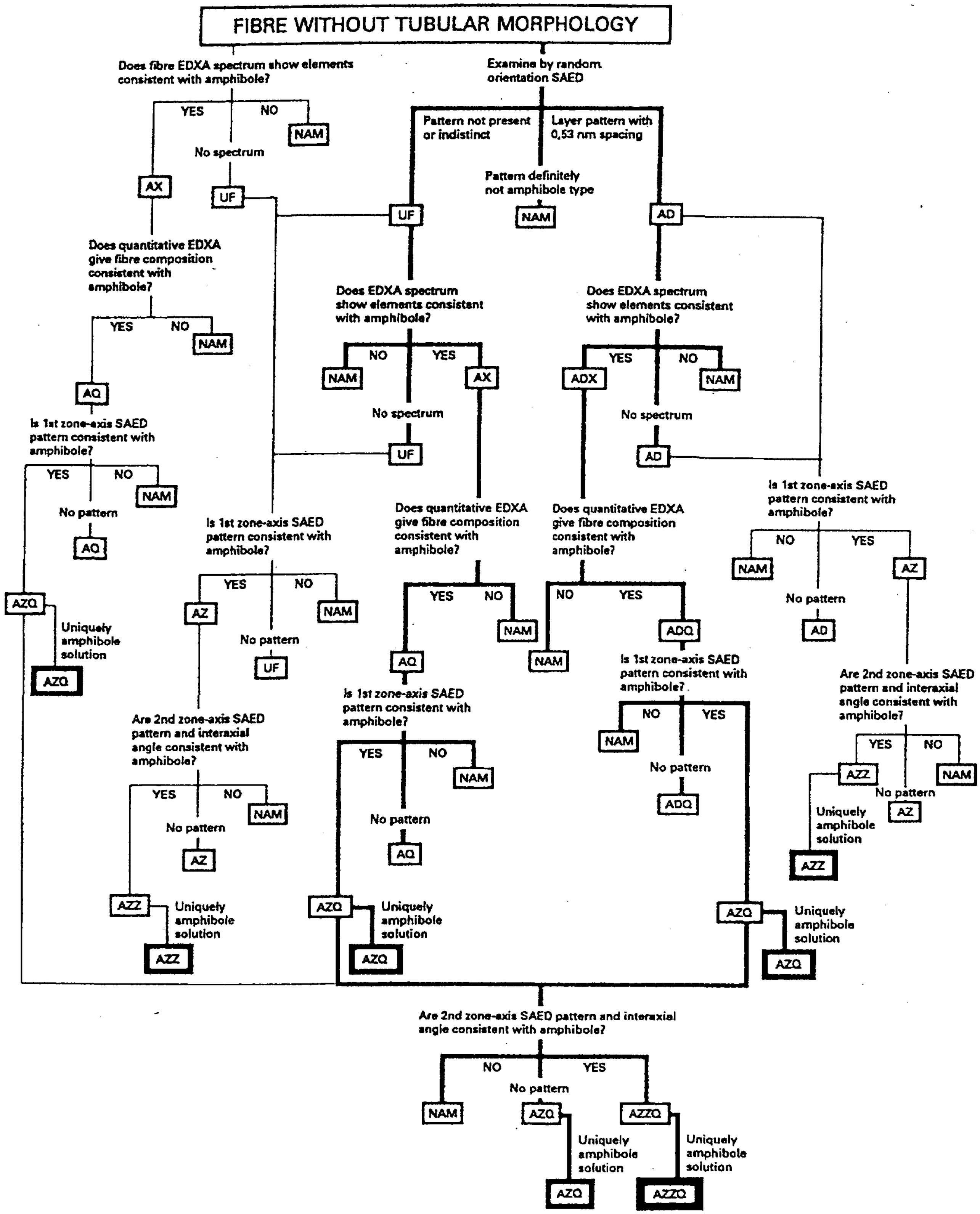


Figure D.4 — Classification chart for fibre without tubular morphology

Annex E
(normative)

Determination of the concentrations of asbestos fibres and bundles longer than 5 μm, and PCM equivalent asbestos fibres

In order to provide increased statistical precision and improved analytical sensitivity for those asbestos fibres and bundles longer than 5 μm, it may be decided to perform additional fibre counting at a lower magnification, taking account only into fibres and bundles within this dimensional range. The result shall be specified as "number of asbestos fibres and bundles longer than 5 μm". For this examination, use a magnification of approximately × 10 000, and continue to assign a morphological code to each structure according to the procedures specified in annex C. Record fibres and bundles only if their lengths exceed 5 μm. Record cluster and matrix components only if their lengths exceed 5 μm.

It may also be decided to provide increased statistical precision and improved analytical sensitivity for fibrous structures longer than 5 μm, with diameters between 0,2 μm and 3,0 μm, which have historically been the basis of risk estimation in the occupational environment (PCM equivalent asbestos fibres). Use a magnification of approximately × 5 000 for this extended fibre count. The result shall be specified as "number of PCM equivalent asbestos fibres". Asbestos structures within this dimensional range do not necessarily incorporate asbestos fibres or bundles longer than 5 μm.

Continue the extended sample examination until 100 asbestos structures have been counted, or until a sufficient area of the specimen has been examined to achieve the desired analytical sensitivity calculated according to table 1. The grid openings examined shall be divided approximately equally between a minimum of two specimen grids.

NOTES

17 The specimen area corresponding to the area of filter examined in the PCM fibre counting methods is 0,785 mm^2, and is equivalent to approximately 100 grid openings of a 200 mesh grid.

18 Some National Standards require that asbestos fibres longer than 2,5 μm, with diameters between 0,2 μm and 3,0 μm be counted. Use a magnification of × 5 000 for counting fibres within these dimensional ranges.

19 The minimum aspect ratio for definition of a fibre in PCM fibre counting methods and in some National Standards is 3/1. Use of a 3/1 aspect ratio is permitted in this International Standard, if this aspect ratio is mentioned in the test report.

The test reports shall include all of the items listed in clause 11.

Annex F
(normative)

Calculation of results

F.1 General

The results should be calculated using the procedures specified below. The results can be conveniently calculated using a computer programme.

F.2 Test for uniformity of distribution of fibrous structures on TEM grids

A chek shall be made using the chi-square test, to determine whether the asbestos structures found on individual grid openings are randomly and uniformly distributed among the grid openings. If the total number found in k grid openings is n, and the areas of the k individual frid openings are designated A_1 to A_k, then the total area of TEM specimen examined is

$$A = \sum_{i=1}^{i=k} A_i$$

The fraction of the total area examined which is represented by the individual grid opening area, p_i, is given by A_i/A. If the structures are randomly and uniformly dispersed over the k grid openings examined, the expected number of structures falling in one grid opening with area A_i is np_i. If the observed number of structures found on that grid opening is n_i, then

$$x^2 = \sum_{i=1}^{i=k} \frac{(n_i - np_i)^2}{np_i}$$

This value shall be compared with significance points of the chi-square distribution, having $(k - 1)$ degrees of freedom. Significance levels lower than 0,1 % may be cause for the sample analysis to be rejected, since this correspond to a very inhomogeneous deposit. If the structure count fails this test, the precision of the result will be uncertain, and if new air samples cannot be collected, additional grid openings may be examined or the sample may be prepared by an indirect method.

F.3 Calculation of the analytical sensitivity

Calculate the required analytical sensitivity S, expressed in number of structures per litre, using the following equation:

$$S = \frac{A_f}{kA_g V}$$

where

- A_f is the area, in square millimetres, of sample collection filter;
- A_g is the area, in square millimetres, of TEM specimen grid opening;
- k is the number of grid openings examined;
- V is the volume of air sampled, in litres.

F.4 Calculation of the mean and confidence interval of the structure concentration

In the structure count made according to this International Standard, a number of grid openings have been sampled from a population of grid openings, and it is required to determine the mean grid opening structure count for the population on the basis of this small sample. The interval about the sample mean which, with 95 % confidence, contains the population mean, is also required.

F.4.1 Calculation of the mean structure concentration

Calculate the mean structure concentration C, expressed in number of structures per litre, using the following equation:

$$C = Sn$$

where

S is the analytical sensitivity, expressed in number of structures per litre;

n is the total number of structures found on all grid openings examined.

F.4.2 Calculation of confidence intervals

The distribution of structures on the grid openings should theoretically approximate to a Poisson distribution. Because of fibre aggregation and size-dependent identification effects, the actual structure counting data often does not conform to the Poisson distribution, particularly at high structure counts. An assumption that the structure counting data are distributed according to the Poisson distribution can therefore lead to confidence intervals narrower than are justified by the data. Moreover, if the Poisson distribution is assumed, the variance is related only to the total number of structures counted. Thus, a particular structure count conducted on one grid opening is considered to have the same confidence interval as that for the same number of structures found on many grid openings. However, the area of sample actually counted is very small in relation to the total area of the filter, and for this reason, structures shall be counted on a minimum of four grid openings taken from different areas of the filter in order to ensure that a representative evaluation of the deposit is made.

At high structure counts, where there are adequate numbers of structures per grid opening to allow a sample estimate of the variance to be made, the distribution can be approximated to a Gaussian, with independent values for the mean and variance. Where the sample estimate of variance exceeds that implicit in the Poissonian assumption, use of Gaussian statistics with the variance defined by the actual data is the most conservative approach to calculation of confidence intervals.

At low structure counts, it is not possible to obtain a reliable sample estimate of the variance, and the distribution also becomes asymmetric but not necessarily Poissonian. For 30 structures and below, the distribution becomes asymmetric enough for the fit to a Gaussian to no longer be a reasonable one, and estimates of sample variance are unreliable. Accordingly, for counts below 31 structures, the assumption of a Poisson distribution shall be made for calculation of the confidence intervals.

F.4.3 Example of calculation of Poissonian 95 % confidence intervals

For total structure counts less than 4, the lower 95 % confidence limit corresponds to less than 1 structure. Therefore, it is not meaningful to quote lower confidence interval points for structure counts of less than 4, and the result shall be recorded as "less than" the corresponding one-sided upper 95 % confidence limit of the Poisson distribution, as follows:

0 structure ≘ 2,99 times the analytical sensitivity

1 structure ≘ 4,74 times the analytical sensitivity

2 structures ≘ 6,30 times the analytical sensitivity

3 structures ≘ 7,75 times the analytical sensitivity

For total counts exceeding 4, the 95 % confidence interval shall be calculated using the values shown in table F.1. Table F.1 gives the upper and lower limits of the two-sided Poissonian 95 % confidence interval for structure counts up to 470.

F.4.4 Example of calculation of Gaussian 95 % confidence intervals

Calculate the sample estimate of variance s^2 using the following equation:

$$s^2 = \frac{\sum_{i=1}^{i=k}(n_i - np_i)^2}{(k-1)}$$

where

n_i is the number of structures on the ith grid opening;

n is the total number of structures found in k grid openings;

p_i is the fraction of the total area examined represented by the ith grid opening;

k is the number of grid openings examined.

If the mean value of the structure count is calculated to be n, the upper and lower values of the Gaussian 95 % confidence interval are given respectively by

$$L_u = \frac{n}{k} + \frac{ts}{\sqrt{k}}$$

and

$$L_l = \frac{n}{k} - \frac{ts}{\sqrt{k}}$$

where

L_u is the upper 95 % confidence limit;

L_l is the lower 95 % confidence limit;

n is the total number of structures in all grid openings examined;

t is the value of Student's test (probability 0,975) for $(k-1)$ degrees of freedom;

s is the standard deviation (square root of sample estimate of variance);

k is the number of grid openings examined.

F.4.5 Summary of procedure for calculation of results

In summary, structure counting data shall be calculated as follows:

No structures detected

The structure concentration shall be reported as less than the concentration equivalent ot the one-sided upper 95 % confidence limit of the Poisson distribution. This is equal to 2,99 times the analytical sensitivity.

From 1 to 3 structures

When 1 to 3 structures are counted, the result shall be reported as less than the corresponding one-sided upper 95 % confidence limit for the Poisson distribution. These are

1 structure ≘ 4,74 times the analytical sensitivity

2 structures ≘ 6,30 times the analytical sensitivity

3 structures ≘ 7,75 times the analytical sensitivity

From 4 to 30 structures

The mean structure concentration and the 95 % confidence intervals shall be reported on the basis of the Poissonian assumption, using the values shown in table F.1.

More than 30 structures

When more 30 structures are counted, both the Gaussian 95 % confidence interval and the Poissonian 95 % confidence interval shall be calculated. The larger of these two intervals shall be used to express the precision of the structure concentration. When the Gaussian 95 % confidence interval is selected for data reporting, the Poissonian 95 % confidence interval shall also be mentioned.

F.5 Calculation of structure length, width, and aspect ratio distributions

The distributions all approximate to logarithmic-normal, and therefore the size range intervals for calculation of the distribution shall be spaced logarithmically. The other characteristics required for the choice of size intervals are that they should allow for a sufficient number of size classes, while still retaining a statistically valid number of structures in each class. Interpretation is also facilitated if each size class repeats at 10 intervals, and if 5 µm is a size class boundary. A ratio from one class to the next of 1,468 satisfies all of these requirements and this value shall be used. The distributions, being approximately logarithmic-normal, when presented graphically, shall be plotted using a logarithmic ordinate scale and a Gaussian abscissa.

F.5.1 Calculation of structure length cumulative number distribution

This distribution allows the fraction of the total number of structures either shorter or longer than a given length to be determined. It is calculated using the following equation:

$$C(P)_k = \frac{\sum_{i=1}^{i=k} n_i}{\sum_{i=1}^{i=P} n_i} \times 100$$

where

$C(P)_k$ is the cumulative number percentage of structures which have lengths less than the upper bound of the kth class;

n_i is the number of structures in the ith length class;

P is the total number of length classes.

F.5.2 Calculation of structure width cumulative number distribution

This distribution allows the fraction of the total number of structures either narrower or wider than a given width to be determined. It is calculated in a similar way to that used in F.5.1, but using the structure widths.

F.5.3 Calculation of structure aspect ratio cumulative number distribution

This distribution allows the fraction of the total number of structures which have aspect ratios either smaller or larger than a given aspect ratio to be determined. It is calculated in a similar way to that used in F.5.1, but using the structure aspect ratios.

Table F.1 — Upper and lower limits of the Poissonian 95 % confidence interval of a count

Structure count	Lower limit	Upper limit	Structure count	Lower limit	Upper limit	Structure count	Lower limit	Upper limit
0	0	3,689[1)]	46	33,678	61,358	92	74,164	112,83
1	0,025	5,572	47	34,534	62,501	93	75,061	113,94
2	0,242	7,225	48	35,392	63,642	94	75,959	115,04
3	0,619	8,767	49	36,251	64,781	95	76,858	116,14
4	1,090	10,242	50	37,112	65,919	96	77,757	117,24
5	1,624	11,669	51	37,973	67,056	97	78,657	118,34
6	2,202	13,060	52	38,837	68,192	98	79,557	119,44
7	2,814	14,423	53	39,701	69,326	99	80,458	120,53
8	3,454	15,764	54	40,567	70,459	100	81,360	121,66
9	4,115	17,085	55	41,433	71,591	110	90,400	132,61
10	4,795	18,391	56	42,301	72,721	120	99,490	143,52
11	5,491	19,683	57	43,171	73,851	130	108,61	154,39
12	6,201	20,962	58	44,041	74,979	140	117,77	165,23
13	6,922	22,231	59	44,912	76,106	150	126,96	176,04
14	7,654	23,490	60	45,785	77,232	160	136,17	186,83
15	8,396	24,741	61	46,658	78,357	170	145,41	197,59
16	9,146	25,983	62	47,533	79,482	180	154,66	208,33
17	9,904	27,219	63	48,409	80,605	190	163,94	219,05
18	10,668	28,448	64	49,286	81,727	200	173,24	229,75
19	11,440	29,671	65	50,164	82,848	210	182,56	240,43
20	12,217	30,889	66	51,042	83,969	220	191,89	251,10
21	13,00	32,101	67	51,922	85,088	230	201,24	261,75
22	13,788	33,309	68	52,803	86,207	240	210,60	272,39
23	14,581	34,512	69	53,685	87,324	250	219,97	283,01
24	15,378	35,711	70	54,567	88,441	260	229,36	293,62
25	16,178	36,905	71	55,451	89,557	270	238,75	304,23
26	16,983	38,097	72	56,335	90,673	280	248,16	314,82
27	17,793	39,284	73	57,220	91,787	290	257,58	325,39
28	18,606	40,468	74	58,106	92,901	300	267,01	335,96
29	19,422	41,649	75	58,993	94,014	310	276,45	346,52
30	20,241	42,827	76	59,880	95,126	320	285,90	357,08
31	21,063	44,002	77	60,768	96,237	330	295,36	367,62
32	21,888	45,175	78	61,657	97,348	340	304,82	378,15
33	22,715	46,345	79	62,547	98,458	350	314,29	388,68
34	23,545	47,512	80	63,437	99,567	360	323,77	399,20
35	24,378	48,677	81	64,328	100,68	370	333,26	409,71
36	25,213	49,840	82	65,219	101,79	380	342,75	420,22
37	26,050	51,000	83	66,111	102,90	390	352,25	430,72
38	26,890	52,158	84	67,003	104,00	400	361,76	441,21
39	27,732	53,315	85	67,897	105,11	410	371,27	451,69
40	28,575	54,469	86	68,790	106,21	420	380,79	462,18
41	29,421	55,622	87	69,684	107,32	430	390,32	472,65
42	30,269	56,772	88	70,579	108,42	440	399,85	483,12
43	31,119	57,921	89	71,474	109,53	450	409,38	493,58
44	31,970	59,068	90	72,370	110,63	460	418,92	504,04
45	32,823	60,214	91	73,267	111,73	470	428,47	514,50

1) The one-sided upper 95 % confidence limit for 0 structures is 2,99.

Annex G
(informative)

Strategies for collection of air samples

G.1 General

An important part of the sampling strategy is a statement of the purpose of the sampling programme. A sufficient number of samples should be collected so that the site is well characterized to the precision and accuracy desired, and also ensure that sample filters appropriately loaded for TEM analysis are obtained from all of the sampling locations.

G.2 Air sample collection in the outdoors environment

Weather conditions restrict the ability to collect satisfactory air samples in the outdoors environment, and whenever possible, sampling should be carried out in low-wind, low-humidity conditions. Detailed records of the weather conditions, windspeed and direction during the sampling period should be made. All available information concerning local topography, and the types and positions of sources should be recorded.

Sequential multipoint sampling is necessary to provide adequate characterization of complex sites and sources. It is recommended that multiple samples are taken upwind and downwind of the site, with a minimum of two samples in the downwind position expected to experience the maximum airborne concentration. The locations of the samplers should be carefully recorded.

G.3 Air sample collection inside buildings

Air samples are often collected inside buildings in which asbestos-containing construction materials are present, in order to determine whether these materials contribute to the asbestos fibre concentration in the building atmosphere. The optimum positions for collection of air samples can only be determined after a complete survey of the building to establish air movement patterns. Multiple samples should be collected in the area where asbestos building materials are present, and control samples should be collected in an adjacent area where no airborne asbestos fibres would be expected. The intakes for air conditioning systems are frequently used as the collection locations for control samples. Whenever possible, static samples should be taken over a period exceeding 4 h during normal activity in the building, at face velocities of between 4 cm/s and 25 cm/s.

Annex H
(informative)

Methods for removal of gypsum fibres

It is common to find fibres of calcium sulfate (gypsum) in airborne particulates collected in buildings and urban environments, and particularly in samples collected where demolition or construction work is in progress. The fibres are readily released when plasters and cement products are disturbed. In some circumstances, particles of calcite or dolomite collected on an air filter can react with atmospheric sulfur dioxide, to form long fibres of gypsum. Gypsum fibres can give rise to high fibre counts by both optical and electron microscopy. The gypsum fibres are often 2 µm to 6 µm long, with aspect ratios greater than 10/1. Sometimes, these fibres appear similar to amphibole asbestos fibres, and in some samples they can be morphologically very similar to chrysotile. In the TEM, the larger fibres have high contrast and at high magnification often exhibit a characteristic mottled appearance which changes under electron beam irradiation. Some gypsum fibres, however, are not easily discriminated from asbestos without examination by EDXA. TEM specimens which contain many such gypsum fibres require an extended examination time in the TEM, because it is necessary to examine each of these fibres by EDXA before it can be rejected.

It is possible to remove gypsum fibres selectively by water extraction. A Jaffe washer (7.3.7), or a condensation washer (7.3.8), should be prepared, but using a water (6.1) as the solvent. The TEM specimens, which have been previously prepared and initially examined in the TEM, should be placed in the washer to allow dissolution of the fibres. If a Jaffe washer is used, the treatment time can be reduced by heating the washer to 90 °C to 100 °C for a few minutes. If a condensation washer is used, the gypsum fibres will be dissolved by treatment for approximately 10 min. The effect of this treatment is to remove the gypsum fibres, leaving carbon replicas (7.3.11) which are readily distinguished from asbestos fibres.

NOTE 20 This procedure should be used only when examination of the untreated TEM specimen grids shows the gypsum fibres to be isolated from any asbestos fibres present. Losses of asbestos fibres may occur if matrices of gypsum and asbestos are exposed to this procedure.

Annex J
(informative)

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ICS 13.040.20

Descriptors: air, quality, air pollution, tests, determination, particle density (concentration), asbestos, microscopic analysis.

Price based on 51 pages

ASBESTOS (bulk) by PLM

9002

various	MW: various	CAS: 1332-21-4	RTECS: C16475000

METHOD: 9002, Issue 2 **EVALUATION: PARTIAL** **Issue 1: 15 May 1989**
Issue 2: 15 August 1994

EPA Standard (Bulk): 1%

PROPERTIES: solid, fibrous, crystalline, anisotropic

SYNONYMS [CAS #]: actinolite [77536-66-4], or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole.

SAMPLING		MEASUREMENT	
BULK SAMPLE:	1 to 10 grams	**TECHNIQUE:**	MICROSCOPY, STEREO AND POLARIZED LIGHT, WITH DISPERSION STAINING
SHIPMENT:	seal securely to prevent escape of asbestos	**ANALYTE:**	actinolite asbestos, amosite, anthophyllite asbestos, chrysotile, crocidolite, tremolite asbestos
SAMPLE STABILITY:	stable	**EQUIPMENT:**	microscope, polarized light; 100-400X dispersion staining objective, stereo microscope: 10-45X
BLANKS:	none required	**RANGE:**	1% to 100% asbestos
ACCURACY		**ESTIMATED LOD:**	<1% asbestos [1]
RANGE STUDIED:	<1% to 100% asbestos	**PRECISION:**	not determined
BIAS:	not determined		
PRECISION:	not determined		
ACCURACY:	not determined		

APPLICABILITY: this method is useful for the qualitative identification of asbestos and the semi-quantitative determination of asbestos content of bulk samples. The method measures percent asbestos as perceived by the analyst in comparison to standard area projections, photos, and drawings, or trained experience. The method is not applicable to samples containing large amounts of fine fibers below the resolution of the light microscope

INTERFERENCES: Other fibers with optical properties similar to the asbestos minerals may give positive interferences. Optical properties of asbestos may be obscured by coating on the fibers. Fibers finer than the resolving power of the microscope (ca. 0.3 µm) will not be detected. Heat and acid treatment may alter the index of refraction of asbestos and change its color.

OTHER METHODS: This method (originally designated as method 7403) is designed for use with NIOSH Methods 7400 (phase contrast microscopy) and 7402 (electron microscopy/EDS). The method is similar to the EPA bulk asbestos method [1].

REAGENTS:

1. Refractive index (RI) liquids for Dispersion Staining: high-dispersion (HD) series, 1.550, 1.605, 1.620.
2. Refractive index liquids: 1.670, 1.680, and 1.700.
3. Asbestos reference samples such as SRM #1866, available from the National Institute of Standards and Technology.*
4. Distilled Water (optional).
5. Concentrated HCl: ACS reagent grade.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sample containers: screw-top plastic vials of 10- to 50-mL capacity.
2. Microscope, polarized light, with polarizer, analyzer, port for retardation plate, 360° graduated rotating stage, substage condenser with iris, lamp, lamp iris, and:
   a. Objective lenses: 10X, 20X, and 40X or near equivalent.
   b. Ocular lense: 10X minimum.
   c. Eyepiece reticle: crosshair.
   d. Dispersion staining objective lens or equivalent.
   e. Compensator plate: ca. 550 nm± 20 nm, retardation: "first order red" compensator.
3. Microscope slides: 75 mm x 25 mm.
4. Cover slips.
5. Ventilated hood or negative-pressure glove box.
6. Mortar and pestle: agate or porcelain.
7. Stereomicroscope, ca. 10 to 45X.
8. Light source: incandescent or fluorescent.
9. Tweezers, dissecting needles, spatulas, probes, and scalpels.
10. Glassine paper or clean glass plate.
11. Low-speed hand drill with coarse burr bit (optional).

SPECIAL PRECAUTIONS: Asbestos, a human carcinogen, should be handled only in an exhaust hood (equipped with a HEPA filter) [2]. Precautions should be taken when collecting unknown samples, which may be asbestos, to preclude exposure to the person collecting the sample and minimize the disruption to the parent material [3]. Disposal of asbestos-containing materials should follow EPA Guidelines [4].

SAMPLING:

1. Place 1 to 10 g of the material to be analyzed in a sample container.
   NOTE: For large samples (i.e., whole ceiling tiles) that are fairly homogenous, a representative small portion should be submitted for analysis. Sample size should be adjusted to ensure that it is representative of the parent material.
2. Make sure that sample containers are taped so they will not open in transit.
3. Ship the samples in a rigid container with sufficient packing material to prevent damage or sample loss.

SAMPLE PREPARATION:

4. Visually examine samples in the container and with a low-magnification stereomicroscope in a hood. (If necessary, a sample may be carefully removed from the container and placed on glassine transfer paper or clean glass plate for examination). Break off a portion of the sample and examine the edges for emergent fibers. Note the homogeneity of the sample. Some hard tiles can be broken, and the edges examined for emergent fibers. If fibers are found, make an estimate of the amount and type of fibers present, confirm fiber type (step 14) and quantify (step 15).
5. In a hood, open sample container and with tweezers remove small, representative portions of the sample.
   1. If there are obvious separable layers, sample and analyze each layer separately.

of each type of material and place on a glass slide.
have thin, inseparable layers, use a scalpel to cut through all the layers
nple. Then cut it into smaller pieces after placing RI liquid on it before
:kness. Alternatively, use a low-speed hand drill equipped with a burr bit
ı hard tiles. Avoid excessive heating of the sample which may alter the
ȝ material.
ample often requires ashing or other specialized preparation, and may
ission electron microscopy for detection of the short asbestos fibers
acteristic of floor tiles.
hard particles, grind it in a mortar. Do not grind so fine that fiber
royed.
tion of the sample in a hood with an appropriate solvent to remove
interfering materials which may be present in the sample. Make
asbestos material removed by this process.
of sample preparation such as acid washing and sodium
e treatment and ashing may be necessary, especially to detect low
of asbestos. If needed, use as described in Reference [1].
RI liquid on the slide, put a small portion of sample in the liquid.
smash small clumps with the flat end of a spatula or probe, producing
les so that better estimates of projected area percentages can be
rticles on the slide so that they are as homogeneous as possible.
of sample should cover the entire area under the cover slip. some
essary to judge the right amount of material to place on the slide. Too
t give sufficient information and too much sample cannot be easily

CONTROL:

h day of operation. Wipe microscope slides and cover slips with lens
efractive index liquids. Record results in a separate logbook.
of the refractive index liquids used once per week of operation. Record
ogbook.
structions for illumination, condenser alignment and other microscope
adjustments prior to each sample set.
entified asbestos species by comparison to standard projections
e detected in a homogeneous sample, examine at least two additional
ng that no asbestos is present.
ion technique might not be able to produce a homogeneous or
slide, prepare a duplicate slide and average the results. Occasionally,
ary greatly, it will be necessary to prepare additional replicate slides and
lts. Prepare duplicate slides of at least 10% of the samples analyzed.
ting.
ples of known asbestos content.
analytical method should participate in the National Voluntary
gram [5] or a similar interlaboratory quality control program. Each
e formal training in polarized light microscopy and its application to
of formal training, laboratory training in asbestos bulk analysis under
estos bulk analyst may be substituted. Owing to the subjective nature
tice is essential in order to remain proficient in estimating projected

asbestos minerals using the optical properties of morphology,

ılike most other analytical
ıd judgment of the analyst.
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letermine the sign of
ı except for crocidolite. If

articular microscope
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ign of elongation.
Examine under plane
interface (i.e., index of
iber morphology for twisted,
ed, ribbon-like morphology
e necessary to cross the
an exact match at 1.550. If
otherwise continue.
Observation of dispersion
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e dispersion staining colors.

mine under plane-polarized
s will be straight, with rigid
ochroic, i.e., it will appear to
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dolite, go to step 15 for

ved the fiber morphology for
n-like or splayed ends. If the
ion staining objective. Blue
d gold and blue colors
is test, go to step 15 for

1.605 HD RI liquid.
olite asbestos. The
e species. Anthophyllite
llel extinction. Actinolite has
ome pleochroism. For all

fragments may also be present. Examine using the central stop dispersion staining objective. Anthophyllite will exhibit central stop colors of blue and gold/gold-magenta; tremolite will exhibit pale blue and yellow; and actinolite will exhibit magenta and golden-yellow colors.

NOTE: In this refractive index range, wollastonite is a common interfering mineral with similar morphology including the presence of cleavage fragments. It has both positive and negative sign of elongation, parallel extinction, and central stop dispersion staining colors of pale yellow and pale yellow to magenta. If further confirmation of wollastonite versus anthophyllite is needed, go to step "j". If any of the above forms of asbestos were confirmed above, go to step 15 for quantitative estimation. If none of the tests above confirmed asbestos fibers, examine the additional preparations and if the same result occurs, report the absence of asbestos in this sample.

j. Wash a small portion of the sample in a drop of concentrated hydrochloric acid on a slide. Place the slide, with cover slip in place, on a warm hot plate until dry. By capillary action, place 1.620 RI liquid under the cover clip and examine the slide. Wollastonite fibers will have a "cross-hatched" appearance across the length of the fibers and will not show central stop dispersion colors. Anthophyllite and tremolite will still show their original dispersion colors.

NOTE: There are alternative analysis procedures to the step-wise approach outlined above which will yield equivalent results. Some of these alternatives are:

i. Perform the initial scan for the presence of asbestos using crossed polars as well as the first-order red compensator. This allows for simultaneous viewing of birefringent and amorphous materials as well as determine their sign of elongation. Some fibers which are covered with mortar may best be observed using this configuration.

ii. Some analysts prefer to mount their first preparation in a RI liquid different than any asbestos materials and conduct their initial examination under plane-polarized light.

iii. If alternative RI liquids are used from those specified, dispersion staining colors observed will also change. Refer to an appropriate reference for the specific colors associated with asbestos in the RI liquids actually used.

QUANTITATIVE ASSESSMENT:

15. Estimate the content of the asbestos type present in the sample using the 1.550 RI preparation. Express the estimate as an area percent of all material present, taking into account the loading and distribution of all sample material on the slide. Use Figure 1 as an aid in arriving at your estimate. If additional unidentified fibers are present in the sample, continue with the qualitative measurement (step 14).

NOTE: Point-counting techniques to determine percentages of the asbestos minerals are not generally recommended. The point-counting method only produces accurate quantitative data when the material on the slide is homogeneous and has a uniform thickness, which is difficult to obtain [6]. The point-counting technique is, recommended by the EPA to determine the amount of asbestos in bulk [1]; however, in the more recent Asbestos Hazard Emergency Response Act (AHERA) regulations, asbestos quantification may be performed by a point-counting or equivalent estimation method [7].

16. Make a quantitative estimate of the asbestos content of the sample from the appropriate combination of the estimates from both the gross and microscopic examinations. If asbestos fibers are identified, report the material as "asbestos-containing". Asbestos content should be reported as a range of percent content. The range reported should be indicative of the analyst's precision in estimating asbestos content. For greater quantities use Figure 1 in arriving at your estimate.

EVALUATION OF METHOD:

The method is compiled from standard techniques used in mineralogy [8-13], and from standard laboratory procedures for bulk asbestos analysis which have been utilized for several years. These tweezers or a spatula before taking the portion of analysis. Alternatively, take small representative portions of each type of material and place on a glass slide.

c. On hard tiles that may have thin, inseparable layers, use a scalpel to cut through all the layers for a representative sample. Then cut it into smaller pieces after placing RI liquid on it before trying to reduce the thickness. Alternatively, use a low-speed hand drill equipped with a burr bit to remove material from hard tiles. Avoid excessive heating of the sample which may alter the optical properties of the material.

   NOTE: This type of sample often requires ashing or other specialized preparation, and may require transmission electron microscopy for detection of the short asbestos fibers which are characteristic of floor tiles.

d. If the sample has large, hard particles, grind it in a mortar. Do not grind so fine that fiber characteristics are destroyed.

e. If necessary, treat a portion of the sample in a hood with an appropriate solvent to remove binders, tars, and other interfering materials which may be present in the sample. Make corrections for the non-asbestos material removed by this process.

   NOTE: Other methods of sample preparation such as acid washing and sodium metaphosphate treatment and ashing may be necessary, especially to detect low concentrations of asbestos. If needed, use as described in Reference [1].

6. After placing a few drops of RI liquid on the slide, put a small portion of sample in the liquid. Tease apart with a needle or smash small clumps with the flat end of a spatula or probe, producing a uniform thickness or particles so that better estimates of projected area percentages can be made. Mix the fibers and particles on the slide so that they are as homogeneous as possible.

   NOTE: An even dispersion of sample should cover the entire area under the cover slip. some practice will be necessary to judge the right amount of material to place on the slide. Too little sample may not give sufficient information and too much sample cannot be easily analyzed.

CALIBRATION AND QUALITY CONTROL:

7. Check for contamination each day of operation. Wipe microscope slides and cover slips with lens paper before using. Check refractive index liquids. Record results in a separate logbook.
8. Verify the refractive indices of the refractive index liquids used once per week of operation. Record these checks in a separate logbook.
9. Follow the manufacturer's instructions for illumination, condenser alignment and other microscope adjustments. Perform these adjustments prior to each sample set.
10. Determine percent of each identified asbestos species by comparison to standard projections (Figure 1) [1]. If no fibers are detected in a homogeneous sample, examine at least two additional preparations before concluding that no asbestos is present.
11. If it appears that the preparation technique might not be able to produce a homogeneous or representative sample on the slide, prepare a duplicate slide and average the results. Occasionally, when the duplicate results vary greatly, it will be necessary to prepare additional replicate slides and average all the replicate results. Prepare duplicate slides of at least 10% of the samples analyzed. Average the results for reporting.
12. Analyze about 5% blind samples of known asbestos content.
13. Laboratories performing this analytical method should participate in the National Voluntary Laboratory Accreditation Program [5] or a similar interlaboratory quality control program. Each analyst should have complete formal training in polarized light microscopy and its application to crystalline materials. In lieu of formal training, laboratory training in asbestos bulk analysis under the direction of a trained asbestos bulk analyst may be substituted. Owing to the subjective nature of the method, frequent practice is essential in order to remain proficient in estimating projected area percentages.

QUALITATIVE ASSESSMENT:

14. Scan the slide to identify any asbestos minerals using the optical properties of morphology,

NOTE: Identification of asbestos using polarized light microscopy is unlike most other analytical methods. The quality of the results is dependent on the skill and judgment of the analyst. This method does not lend itself easily to a step-wise approach. Various procedures devised by different analysts may yield equivalent results. The following step-wise procedure repeatedly utilizes the sample preparation procedure previously outlined.

a. Prepare a slide using 1.550 HD RI liquid. Adjust the polarizing filter such that the polars are partially crossed, with ca. 15° offset. Scan the preparation, examining the morphology for the presence of fibers. If no fibers are found, scan the additional preparations. If no fibers are found in any of the preparations, report that the sample does not contain asbestos, and stop the analysis at this point.

b. If fibers are found, adjust the polarizing filter such that the polars are fully crossed. If all of the fibers are isotropic (disappear at all angles of rotation) then those fibers are not asbestos. Fibrous glass and mineral wool, which are common components of suspect samples, are isotropic. If only isotropic fibers are found in the additional preparations, report no asbestos fibers detected, and stop the analysis.

c. If anisotropic fibers are found, rotate the stage to determine the angle of extinction. Except for tremolite-actinolite asbestos which has oblique extinction at 10-20°, the other forms of asbestos exhibit parallel extinction (Table 1). Tremolite may show both parallel and oblique extinction.

d. Insert the first order red compensator plate in the microscope and determine the sign of elongation. All forms of asbestos have a positive sign of elongation except for crocidolite. If the sign of elongation observed is negative, go to step "g."

   NOTE: To determine the direction of the sign of elongation on a particular microscope configuration, examine a known chrysotile sample and note the direction (NE-SW or NW-SE) of the blue coloration. Chrysotile has a positive sign of elongation.

e. Remove the first-order red compensator and uncross the polarizer. Examine under plane polarized light for blue and gold-brown Becke colors at the fiber-oil interface (i.e., index of refraction match). Becke colors are not always evident. Examine fiber morphology for twisted, wavy bundles of fibers which are characteristic of chrysotile. Twisted, ribbon-like morphology with cellular internal features may indicate cellulose fibers. It may be necessary to cross the polars partially in order to see the fibers if the index of refraction is an exact match at 1.550. If the fibers appear to have higher index of refraction, go to step "h," otherwise continue.

f. Identification of chrysotile. Insert the dispersion staining objective. Observation of dispersion staining colors of blue and blue-magenta confirms chrysotile. Cellulose, which is a common interfering fiber at the 1.550 index of refraction, will not exhibit these dispersion staining colors. If chrysotile is found, go to step 15 for quantitative estimation.

g. Identification of crocidolite. Prepare a slide in 1.700 RI liquid. Examine under plane-polarized light (uncrossed polars); check for morphology of crocidolite. Fibers will be straight, with rigid appearance, and may appear blue or purple-blue. Crocidolite is pleochroic, i.e., it will appear to change its color (blue or gray) as it is rotated through plane polarized light. Insert the dispersion staining objective. The central stop dispersion staining color are red magenta and blue magenta, however, these colors are sometimes difficult to impossible to see because of the opacity of the dark blue fibers. If observations above indicate crocidolite, go to step 15 for quantitative estimation.

h. Identification of amosite. Prepare a slide in 1.680 RI liquid. Observed the fiber morphology for amosite characteristics: straight fibers and fiber bundles with broom-like or splayed ends. If the morphology matches amosite, examine the fibers using the dispersion staining objective. Blue and pale blue colors indicate the cummingtonite form of amosite, and gold and blue colors indicate the grunerite form of amosite. If amosite is confirmed by this test, go to step 15 for quantitative estimation, otherwise continue.

i. Identification of anthophyllite-tremolite-actinolite. Prepare a slide in 1.605 HD RI liquid. Examine morphology for comparison to anthophyllite-tremolite-actinolite asbestos. The refractive indices for these forms of asbestos vary naturally within the species. Anthophyllite can be distinguished from actinolite and tremolite by its nearly parallel extinction. Actinolite has a light to dark green color under plane-polarized light and exhibits some pleochroism. For all fragments may also be present. Examine using the central stop dispersion staining objective. Anthophyllite will exhibit central stop colors of blue and gold/gold-magenta; tremolite will exhibit pale blue and yellow; and actinolite will exhibit magenta and golden-yellow colors.

NOTE: In this refractive index range, wollastonite is a common interfering mineral with similar morphology including the presence of cleavage fragments. It has both positive and negative sign of elongation, parallel extinction, and central stop dispersion staining colors of pale yellow and pale yellow to magenta. If further confirmation of wollastonite versus anthophyllite is needed, go to step "j". If any of the above forms of asbestos were confirmed above, go to step 15 for quantitative estimation. If none of the tests above confirmed asbestos fibers, examine the additional preparations and if the same result occurs, report the absence of asbestos in this sample.

j. Wash a small portion of the sample in a drop of concentrated hydrochloric acid on a slide. Place the slide, with cover slip in place, on a warm hot plate until dry. By capillary action, place 1.620 RI liquid under the cover clip and examine the slide. Wollastonite fibers will have a "cross-hatched" appearance across the length of the fibers and will not show central stop dispersion colors. Anthophyllite and tremolite will still show their original dispersion colors.

NOTE: There are alternative analysis procedures to the step-wise approach outlined above which will yield equivalent results. Some of these alternatives are:

i. Perform the initial scan for the presence of asbestos using crossed polars as well as the first-order red compensator. This allows for simultaneous viewing of birefringent and amorphous materials as well as determine their sign of elongation. Some fibers which are covered with mortar may best be observed using this configuration.

ii. Some analysts prefer to mount their first preparation in a RI liquid different than any asbestos materials and conduct their initial examination under plane-polarized light.

iii. If alternative RI liquids are used from those specified, dispersion staining colors observed will also change. Refer to an appropriate reference for the specific colors associated with asbestos in the RI liquids actually used.

QUANTITATIVE ASSESSMENT:

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    NOTE: Point-counting techniques to determine percentages of the asbestos minerals are not generally recommended. The point-counting method only produces accurate quantitative data when the material on the slide is homogeneous and has a uniform thickness, which is difficult to obtain [6]. The point-counting technique is, recommended by the EPA to determine the amount of asbestos in bulk [1]; however, in the more recent Asbestos Hazard Emergency Response Act (AHERA) regulations, asbestos quantification may be performed by a point-counting or equivalent estimation method [7].

16. Make a quantitative estimate of the asbestos content of the sample from the appropriate combination of the estimates from both the gross and microscopic examinations. If asbestos fibers are identified, report the material as "asbestos-containing". Asbestos content should be reported as a range of percent content. The range reported should be indicative of the analyst's precision in estimating asbestos content. For greater quantities use Figure 1 in arriving at your estimate.

EVALUATION OF METHOD:

The method is compiled from standard techniques used in mineralogy [8-13], and from standard laboratory procedures for bulk asbestos analysis which have been utilized for several years. These

Assurance Program samples since 1982 [1,5]. However, no formal evaluation of this method, as written, has been performed.

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METHOD WRITTEN BY:

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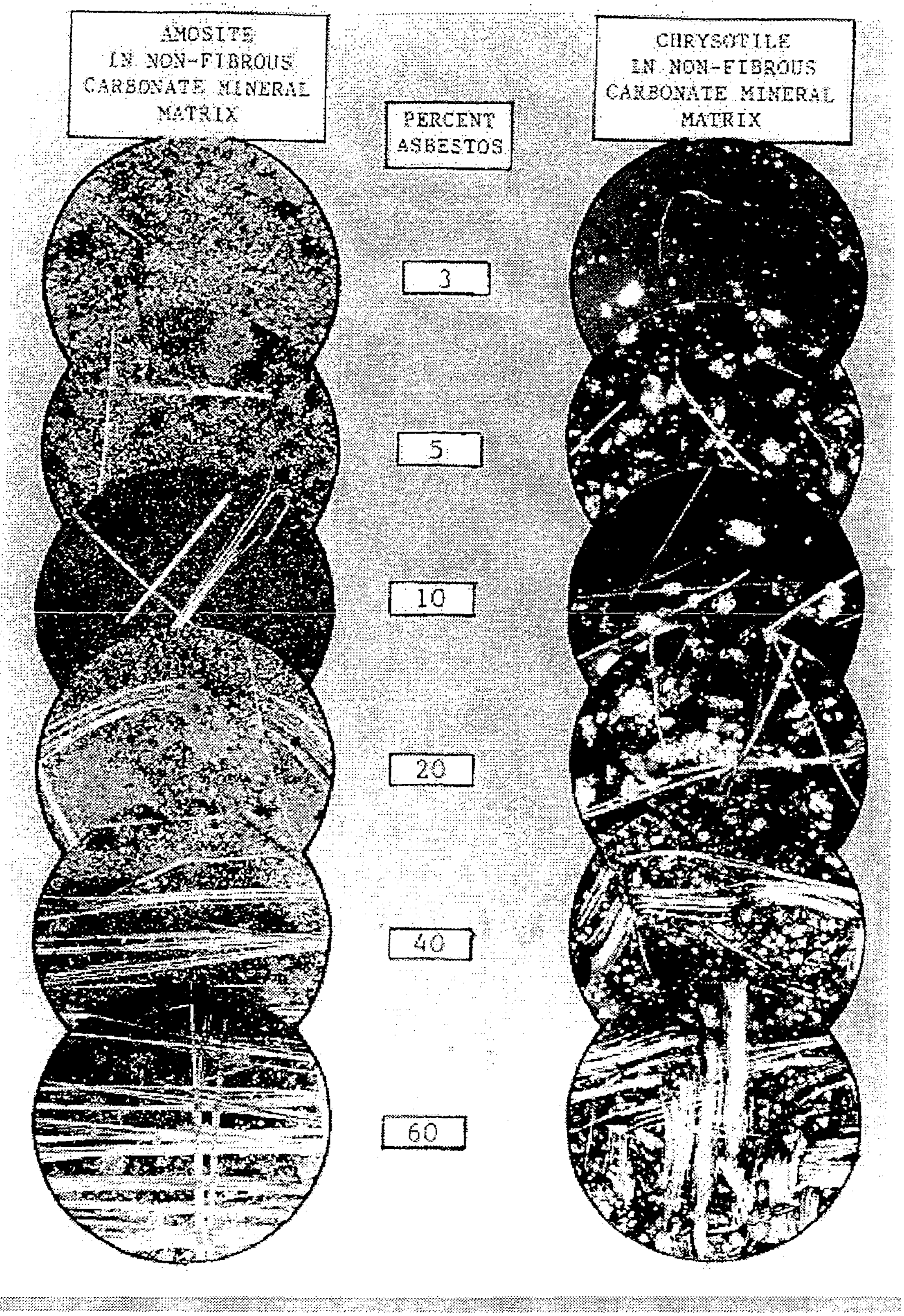

Figure 1. Percent estimate comparator

Table 1. Optical Properties of Asbestos Fibers

Mineral	Morphology and Color	Refractive Index (Approximate Values) ⊥ to Elongation	Refractive Index (Approximate Values) ∥ to Elongation	Birefringence
Chrysotile	Wavy fibers with kinks. Splayed ends on larger bundles. Colorless to light brown upon being heated. Nonpleochroic. Aspect ratio typically >10:1.	1.54	1.55	0.002 - 0.014
Cummingtonite-Grunerite (Amosite)	Straight fibers and fiber bundles. Bundle ends appear broom-like or splayed. Colorless to brown upon heating. May be weakly pleochroic. Aspect ratio typically >10:1.	1.67	1.70	0.02 - 0.03
Crocidolite (Riebeckite)	Straight fibers and fiber bundles. Longer fibers show curvature. Splayed ends on bundles. Characteristic blue color. Pleochroic. Aspect ratio typically >10:1.	1.71	1.70	0.014 - 0.016 Interference colors may be masked by blue color.
Anthophyllite	Straight fibers and fiber bundles. Cleavage fragments may be present. Colorless to light brown. Nonpleochroic to weakly pleochroic. Aspect ratio generally <10:1.	1.61	1.63	0.019 - 0.024
Tremolite-Actinolite	Straight and curved fibers. Cleavage fragments common. Large fiber bundles show splayed ends. Tremolite is colorless. Actinolite is green and weakly to moderately pleochroic. Aspect ratio generally <10:1.	1.60 - 1.62 (tremolite) 1.62 - 1.67 (actinolite)	1.62 - 1.64 (tremolite) 1.64 - 1.68 (actinolite)	0.02 - 0.03

Table 1. Optical Properties of Asbestos Fibers (Continued)

			Central Stop Dispersion Staining Colors		
Mineral	Extinction	Sign of Elongation	RI Liquid	⊥ to Vibration	∥ to Vibration
Chrysotile	Parallel to fiber length	+ (length slow)	1.550 HD	Blue	Blue-magenta
Cummingtonite-Grunerite (Amosite)	Parallel to fiber length	+ (length slow)	1.670 Fibers subjected to high temperatures will not dispersion-stain.	Red magenta to blue	Yellow
Cummingtonite Grunerite			1.680 1.680	pale blue blue	blue gold
Crocidolite (Riebeckite)	Parallel to fiber length	- (length fast)	1.700 1.680	Red magenta yellow	Blue-magenta pale yellow
Anthophyllite	Parallel to fiber length	+ (length slow)	1.605 HD 1.620 HD	Blue Blue-green	Gold to gold-magenta Golden-yellow
Tremolite-Actinolite	Oblique - 10 to 20° for fragments. Some composite fibers show ∥ extinction.	+ (length slow)	1.605 HD	Pale blue (tremolite) Yellow (actinolite)	Yellow (tremolite) Pale yellow (actinolite)

HD = high-dispersion RI liquid series.